

Chemical & Process Engineering

Vol. 34, No. 9

CONTENTS

SEPTEMBER 1953

TOPICS OF THE MONTH	267	THE GASIFICATION OF COAL	291
Chemical Engineering Reviews:		ULTRASONIC TESTING OF WELDED CHEMICAL PLANT	293
ION EXCHANGE by J. M. Hutcheon, B.Sc.	271	PLANT AND EQUIPMENT:	
VACUUM TECHNIQUE by S. L. Martin, M.Sc., A.R.C.S., D.I.C., F.R.I.C.	276	Line blind valves; Rod mills; Hydrostatic contents control; TV for remote water gauge indication; Flameproof barrier box ...	294
BATCH VERSUS CONTINUOUS DISTILLATION—2 by S. R. M. Ellis, Ph.D., F.R.I.C., A.M.I.Chem.E., and J. T. Shelton, B.Sc.	280	CHEMICAL ENGINEERING INVENTION	296
ACETIC ANHYDRIDE FROM ACETONE	283	WORLD NEWS:	
HYDRODESULPHURISATION	285	From Great Britain, Australia, New Zealand, South Africa, Chile, Cuba, India, Norway, Turkey, Canada, Brazil, Mexico, Colombia, Japan, United States	297
YEAST FROM MOLASSES	288	MEETINGS	302
NEW STANDARDS	290		

Subscription rates: 1 year 6 dollars, 3 years 15 dollars, paid in advance

England: Leonard Hill Limited, Stratford House, 9 Eden Street, London, N.W.1

U.S.A.: British Publications Inc., 30, East 60th St., New York, 22

Topics of the Month

New sulphur-from-oil plants

ALTHOUGH the immediate shortage of sulphur has long passed, the projects for increasing home output put in hand or planned under the stimulus of the crisis in 1950-51 are now coming into production, to the lasting benefit of the British economy. The big oil companies were quick off the mark in planning plants which would simultaneously purify their products and add to indigenous sulphur supplies. Shell were the first to go on stream with their recovery plant at Stanlow refinery which turns out 10,000 tons p.a. of 99.9% pure rock sulphur. This plant was described in *CHEMICAL & PROCESS ENGINEERING* in July 1952. Last month the Esso Petroleum Co. Ltd. announced that their sulphur recovery plant at Fawley refinery had started operation. This has a capacity of more than 14,000 tons p.a. of 99.5% pure rock sulphur.

Next year two more sulphur-from-petroleum plants should go on stream. Anglo-Iranian are building one at their Grangemouth refinery which, like Shell's and Esso's, will process gases from a catalytic cracker. The three plants appear to be broadly similar in that the gases from the cracker are washed with an amine solution (diethanolamine) to remove the hydrogen sulphide, which is burnt with air under controlled conditions and by reaction over a catalyst to yield liquid sulphur which is pumped to 'boxes' where it solidifies to a bright yellow 'rock.' Anglo-Iranian do not

give an estimate of their probable sulphur production, but it is a fair guess that it will be in the region of 10,000 tons p.a.

The second refinery sulphur plant scheduled to start up next year will again be at Stanlow. It will cost £1 million, consist almost entirely of British materials and will produce between 10,000 and 15,000 tons p.a. Technically it is more interesting than the other three plants because it will utilise for the first time an entirely new process called "hydrodesulphurisation." This process, described fully in an article in this issue of *CHEMICAL & PROCESS ENGINEERING*, has been developed in the Amsterdam laboratories of the Royal Dutch/Shell Group. It differs from the conventional vapour-phase procedure in that hydrodesulphurisation is effected by the 'trickle' technique, so called because the feedstock trickles in liquid form over the bed of catalyst in the presence of hydrogen under pressure. In practice the oil, after meeting a circulating stream of hydrogen, is heated and the mixture passed over a fixed bed of catalyst (cobalt molybdena on alumina). Reactor pressures are of the order of 300 to 750 p.s.i.g., and reactor temperatures 650 to 730°F. according to the nature of the feedstock. A sulphur reduction of 85 to 90% can be effected with very little attack of the cracked or highly aromatic components present in the feedstock. Since the feedstock does not have to be vaporised, the new process results in considerable economies in heat transfer equipment and fuel, as well as in hydrogen com-

pressors, pipelines and valves. It is anticipated that regeneration of the catalyst will be infrequent, if required at all, and that on-stream periods of twelve months will be possible in most cases.

When these four plants are working, probably by the end of 1954, the British oil-refining industry will be producing something like 45,000 tons of sulphur each year, a very useful addition indeed to our supplies, particularly because of the high purity of the product.

The quest for absolute zero

THE application of low temperatures in industry, for producing oxygen and the inert gases and in other directions, is sufficient reason for others than academic scientists to commemorate the centenary of the birth of Heike Kammerlingh Onnes on September 21. Onnes scored a resounding success when he liquefied helium after Dewar, Travers and Olszewski had failed, and he was awarded the Nobel Prize in 1913.

Son of a tile manufacturer of Gröningen, Onnes was a prize essayist at Utrecht in the field of physical chemistry before spending two years at Heidelberg under Bunsen and Kirchhoff in their hey-days in spectrum analysis. Onnes returned to Holland for a spell at Delft, and then in 1882 became professor of experimental physics at Leyden, to stay there for the rest of his career. The cryogenic laboratory there, which became a research centre of international reputation, is now the Kammerlingh Onnes Laboratory of the University of Leyden in tribute to this pioneer who developed gauges for high pressures and apparatus for measuring low temperatures, as well as discovering the super-conductivity of metals at such temperatures.

On the occasion of his centenary we realise today how this quest for the absolute zero proved such a fascinating chapter of grim endeavour. Compression and cooling of gases opened the story, with Faraday liquefying chlorine and so neatly replying to Dr. Paris, who had chided him for using greasy vessels: 'Dear sir, the oil you noticed yesterday turned out to be liquid chlorine. Yours faithfully, Michael Faraday.' Only two years elapsed before Thilorier saw his liquefied carbon dioxide explode as the cast-iron vessel killed a demonstrator in a lecture room full of students. Raoul Pictet with his 'cascade system' of stepping down in temperature began the first definite series of experiments designed to liquefy most gases, but not the so-called 'permanent gases' like hydrogen and helium. Andrews with his postulate of a 'critical temperature' above which a gas could not be liquefied, and then the Joule-Thomson effect, both helped to fix the basis for success with permanent gases, particularly when it was further shown that for hydrogen and helium the normal Joule-Thomson effect was reversed until the gases had been initially cooled below the 'inversion temperature.' Dewar's vacuum flask designed to keep out heat rather than maintain hot liquids within; the work of Hampson, Linde, and of Olszewski's associate, Sigmund von Wroblewski, a Polish patriot who was banished to Siberia—all such work contributed to the cause. Then in 1908 after years of effort Onnes took helium he had prepared from monazite sand, compressed the gas and cooled it with liquid hydrogen to 15 to 16°K., and allowed it to expand. He used 20 l. of precious liquid hydrogen and, when the last bottle was used and the helium had gone through the cycle 20 times, liquid helium was won. Onnes made five unsuccessful attempts to solidify helium, but this had to wait until Keesom, his successor at Leyden, succeeded.

Dust emission from cement works

THE problem of dust emission from cement works in England and Wales is referred to in the annual report of the Alkali Inspectors, published at the end of July.* The high demand for cement of the last two years has continued unabated, the report states, and kilns have been shut down only when necessary for essential repairs and adjustments. Considering the intensive production, complaint, especially on Thames-side, was probably inevitable. Close attention has been paid and the Deputy Chief Inspector has paid a personal visit to every cement works in England and Wales during the year.

Failure of electrical precipitators used for the de-dusting of kiln gases has been a matter for grave concern. Most of the trouble has been of mechanical origin, due chiefly to corrosion of parts within the precipitator. There have been discussions with the industry as to the means to be taken to reduce to the minimum condensation and the consequent corrosion and all the troubles that such entails.

Progress in dealing with low-level dust emissions from grinding, bagging and so forth continues at a reasonably satisfactory rate. The next few years should see all works equipped to deal with this problem, even the small, remote works against which there is no complaint.

Considerable attention continues to be paid to Thames-side, where about half the cement in the country is produced. The Ministry is in close touch with the local authorities and it had been hoped that this report would be able to record further progress. The failure of a number of precipitators has, however, led to deterioration of a temporary nature, fortunately offset to some extent by improvements resulting from progressive reduction of emissions at low level. Complaint from the Medway region has been much less than in 1951.

The report adds: 'The painfully-gained experience of the factors militating against continued steady operation of electrical precipitators should result in better operation of the rebuilt units and prevent more recently erected units going the way of the others. Meantime, dust emission from the affected kilns is being kept as low as possible by careful attention to the chain systems.'

The report also gives a general review of all the work carried out by the inspectors during 1952. In this period they paid 3,786 visits to works in England and Wales at which heavy chemical and allied operations are carried on, the effects from which are liable to cause danger or nuisance in the areas in which they are situated. The number of works registered under the Alkali, etc., Works Regulation Act, 1906, in 1952 was 964, involving the operation of 1,785 separate processes. This was a decrease of 7 in the number of works, but an increase of 12 in the number of processes operated, compared with the previous year. In view of the increasing size and complexity of the works which have to be inspected, an additional inspector was recruited, who will operate from Manchester.

In Scotland 83 works which had 122 manufacturing processes requiring inspection were registered, a net increase of 9 works and 11 processes. Visits of inspection to registered works were carried out on 211 occasions and 207 visits were made to non-registered works. The decline in sulphuric acid production in Scotland noted in the previous year continued, but at a lesser rate.

* The 89th Annual Report on Alkali, etc., Works by the Chief Inspectors, 1952, presented to the Minister of Housing and Local Government and to the Secretary of State for Scotland (H.M.S.O., 2s. 6d.).

So you want to be a chemical engineer?

WHAT is a chemical engineer? What does he do and where does he work? These questions and their variations are regularly addressed to the Institution of Chemical Engineers by schoolboys, schoolmasters and youth employment officers. This evidence of a widespread interest in the profession has encouraged the Institution to publish a 16-page brochure, 'Careers in Chemical Engineering,' designed to answer most of the questions that a youth on the brink of choosing a career would ask.

Aiming at lucidity and brevity, the authors of the brochure have deliberately chosen simple language, yet they have nevertheless given a detailed sketch of their subject. First there is a brief historical introduction. Then the misapprehension that chemical engineering is just a mixture of chemistry and mechanical engineering is disposed of and the concept of unit processes and unit operations is explained. There follows a description of the work chemical engineers do, operating and designing plants and so forth. The spheres of employment are next explained on the basis of the 1950 survey of the Ministry of Labour. Of especial interest to the prospective entrant is the statement that considerably more chemical engineers are wanted than are being trained. Finally, there is a summary of the ways in which chemical engineers are trained—universities (seven have chemical engineering courses), technical colleges (about 20 have courses), post-graduate courses and so on. This part should interest more than schoolboys and teachers because in industry itself there is lack of knowledge of the means for training now available. This information will shortly be supplemented by a list of courses now being prepared by the Institution.

Several hundreds of these brochures are being issued free to schools, colleges, youth employment officers and so on and they should certainly stimulate interest and, it is to be hoped, instil into many lads the ambition to become chemical engineers. One addition that could have been made is a note on financial rewards.

The brochure has been financed entirely by the Institution, but it is hoped that there will be demands from industry for copies for which a charge of 2s. each will be made to cover some of the costs of production.

Water-repelling greases

LUBRICATING greases are made by adding absorbent materials to oils. However, many of these absorbent materials, such as silica gel, have an even greater affinity for water than for oil, and the greases gradually disintegrate when they come in contact with water.

There are a number of ways to make them repel water, but an easier and cheaper process has recently been developed by the National Research Council. The inventors are Drs. I. E. Puddington and A. F. Sirianni of N.R.C.'s Division of Applied Chemistry.

In earlier processes, the absorbent material was treated with a water repellent before the oil was added. In the N.R.C. process, a small amount of drying oil is first added to the lubricating oil; then the absorbent material is added, and all the drying oil is promptly adsorbed on the surfaces of the absorbent particles. When this mixture is heated, the drying oil forms a water-repellent coating over the particles, to produce a water-repelling grease. This is much simpler and more economical than the previous methods. Details and licensing arrangements are available from Canadian Patents & Development Ltd., Ottawa.

Indian plant makers' teething troubles

FORMIDABLE competition from foreign firms is hampering Indian chemical plant manufacturers in their efforts to develop. One of the troubles of the Indian firms, apparently, is that they do not get much support either from the Indian Government or from the governments of the various states. Imports from foreign manufacturers are encouraged—and seemingly preferred—even for such items as ordinary steam-jacketed vessels and vacuum distillation units, which are fabricated in India at 20 to 30% lower cost. Another sore point with Indian manufacturers is that the Indian Government's five-year plan makes no mention of the need to develop a fully-fledged chemical engineering industry.

But, according to an article by D. Datta in the Indian journal, *Chemical Age*, in which these points are raised, there are at present only about a dozen firms in India who specialise in fabricating chemical plant and equipment, and they meet only a fraction of the total demand. This being the case, it is nonsense to suppose that India can do without foreign chemical plant.

The author of the article also concedes that many of the Indian firms do not have suitably qualified technical staff experienced in making specialised equipment. Confidence in the ability of such firms to supply satisfactory plant is especially shaken when they quote at rock-bottom prices in an effort to draw buyers and then deliver faulty equipment.

In some cases, quotations and drawings submitted to specifications show that the supplier does not know even basic chemical engineering principles!

To turn to the other side of the picture, a complaint made by the Indian fabricators is that users of plant and equipment in India are very reluctant to disclose their exact requirements, so that the fabricator does not have the detailed specifications necessary to carry out a reliable job. The client is sometimes not willing even to reveal whether the units required are intended to handle liquids, solids or gases, nor their mixing time, temperature and duration of treatment. The result is that quotations and drawings have to be submitted on speculation.

If this is true, Indian plant manufacturers certainly have a reasonable complaint. Their first step, obviously, is to remove the suspicion that gives rise to this unco-operative attitude. Clearly, foreign firms enjoy the confidence of their customers, otherwise they would not be able to satisfy their needs.

The charge of incompetence cannot be levelled at all fabricating firms in India. Some have highly-trained staff and a technical laboratory of their own. In some cases an arrangement is made with institutions whereby the process is worked out on pilot-plant scale as a basis for preparing the quotation for the full-scale plant.

Fluoridation of water

ALLIED to the chemical modification or fortification of foods to preserve them or improve their nutritional value, is the practice of adding chemicals for medicinal purposes. In 1948, the Medical Research Council in Britain recommended that potassium iodide be added to table salt as a prophylactic measure against goitre and since then iodised salt has been available in the shops. This recommendation was only made after other countries had been adding iodine to salt for years. Similar caution was shown

towards the practice of adding sodium fluoride to water to reduce tooth decay which has proved markedly effective in the United States. Now, however, a British mission has returned from studying fluoridation in the U.S. with the recommendation that it should be done in Britain. However, before it is universally adopted, it is suggested that area investigations should be made.

In their report, 'The fluoridation of domestic water supplies in North America as a means of controlling dental caries,' published by the Stationery Office (5s. net), the mission observes that fluoridation is not a cure for tooth decay, nor is it known whether it will retard the progress of existing decay. It is a long-term measure that in time will substantially reduce the incidence of decay in the population. Fluoridation of water is preferable to the topical application of sodium fluoride to the teeth or the use of tablets for the dosing of individual supplies. The latter might be risky, whereas with a properly designed plant and controls there is no danger of adding a toxic overdose at the water plant. Mottling of teeth is not considered to be a hazard and no other dangers to health are considered likely to arise from fluoridation.

In the U.S. there are over 35 million people in 45 states receiving fluoridated water.

Equipment is stated to cost between \$400 (about £145) and \$5,000 (about £1,800), depending on the type and size of the 'feeder' required. The actual cost of fluoridation for Detroit, with a population of about 2.5 million, was estimated to be 3 cents (about 2½d.) per head per annum.

Sea water still for crop irrigation

RECENTLY there has been much discussion of methods and techniques for demineralising salt water. In our May issue, we reported that the Organisation for European Economic Co-operation is sponsoring research on this problem and in July we presented the second part of a comprehensive survey of demineralising methods which we started in April. Now comes news of an essentially simple solar still designed for producing irrigation water in tropical countries. This is envisaged as a modification of a life-raft type solar still, designed by Dr. Maria Telkes of the Massachusetts Institute of Technology and described in a recent issue of *Industrial & Engineering Chemistry*.

The life-raft still, now standard equipment, consists essentially of an inflatable transparent envelope made of *Vinylite* sheet material, supporting a black porous pad saturated with sea water. The still floats on the water, the rays of the sun transmitted by the envelope heat the sea water, and the vapour condenses on the cold surfaces and is collected in the container at the bottom of the still. During a test this device operated with a 50 to 60% efficiency and the temperature of the porous pad was around 150°F. during midday.

Dr. Telkes suggests that tubular modifications of this still could be mounted on posts and the distilled water used to irrigate the soil below. In this case a feeding conduit could be provided to supply the absorbent, porous pad with sea water, flowing at a slow, continuous rate and flushing out any solid residue during the night. In tropical regions the stills could supply $\frac{1}{2}$ in. of water daily over the area occupied by them—equivalent to a monthly rainfall of 10 in. If a monthly rainfall of 1 to 2 in. be considered sufficient for agricultural purposes, solar stills might water an area five to ten times larger than the area occupied by them. The success of this arrangement is primarily an economic

problem. Solar stills of this type, mounted on sufficiently high supports, would not interfere with agricultural operations. The shadow cast by the still travels during the day and would not impede the growth of the plants. The partial shading effect may even be desirable, protecting the plants from the fierce tropical sun.

Argentina builds its chemical industry

CHEMICAL industry in Argentina is scarcely more than a generation old, yet its 40 companies supply a large portion of the nation's chemical requirements. One of the milestones of the road towards a real chemical industry was the erection of a petroleum cracking plant by the government in 1925. Another, which set off a sort of chain reaction, was the beginning of the production of cellulose from straw. In this application of the Pomilio process was the beginning of Argentina's electrolytic alkali industry and a varied array of descendents—hydrochloric acid, hypochlorites, metallic chlorides, sulphur chloride, chloroform and, finally, the first synthetic ammonia plant in South America.

Other important chemical advances followed: contact sulphuric acid and sodium silicate (1929), large-scale production of tartaric acid (1936), rayon (1937), acetic acid and derivatives (1941), hydrogen peroxide (1940) and butanol and acetone by fermentation. Production of nitric acid and fuming sulphuric acid for explosives was begun by the government, and 1945 marked the first blast-furnace production of pig iron in the Argentine. Isopropyl alcohol, from cracking gases, can be considered the starting point, in 1944, of a petrochemical industry.

From 1935 to 1941, the number of plants devoted to chemical manufacturing, including pharmaceuticals and protective coatings, increased from 387 to 537. With chemical production in 1943 taken as a base and assigned the value 100, 1951 production was 148.

At the end of World War 2 some of the greatly expanded war-time chemical lines were unable to compete economically against foreign products in a peace-time economy. Exchange control enforced by the government improved this situation somewhat, but high cost of domestic production of some intermediates has hindered progress of other industries requiring these materials. Shortage of exchange, leading to restricted importation, hit the chemical industry hard in another way—lack of critical raw materials and new machinery.

In spite of these factors, however, the Argentine chemical industry has continued to grow.

Another healthy sign, according to an article by W. H. Shearon in *Chemical and Engineering News*, is that the Argentine schools of higher education are supplying most of the chemically-trained graduates required by industry. When new plants are built by foreign-owned companies, they may yet be started up by foreign engineers, but operation is later turned over to Argentine-trained personnel. In some cases, the Argentines themselves put the plants into operation.

Only one school in Argentina, in Santa Fé, gives a degree in chemical engineering. A number of universities offer degrees in chemistry, with the University of Buenos Aires—the only one offering a doctorate in chemistry—heading the list in output of graduates. Generally speaking, technical training in Argentine universities is considered by industry to be quite good, although the amount of practical work which can be done by the student while in school is limited by inadequate laboratory facilities.

ION EXCHANGE

Water and effluent treatment; sugar refining; pharmaceutical processing; research applications; materials; new techniques; theory

By J. M. Hutcheon, B.Sc.

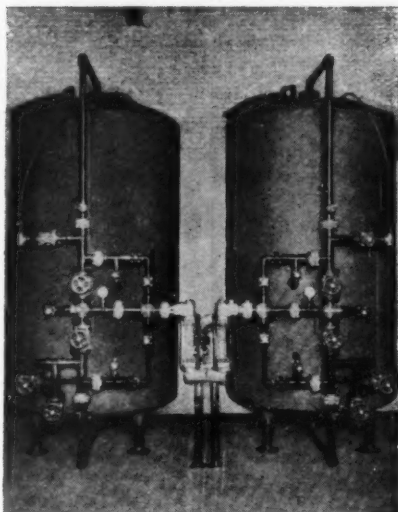
ION exchange is now well established as a unit operation of chemical engineering, particularly adapted to the removal of ions present in solution at concentrations of the order of 1% or less, for which purpose it can often be made very selective. It is often appropriate where the ion concerned is either very valuable or very objectionable and its staple applications are to water treatment, sugar manufacture and effluent treatment. New developments in these fields will be considered together with other applications in this review, which covers the approximate period from the last review¹ in this journal to date. Full bibliographies covering most of the period will be found in reviews^{2, 3} published in January 1952 and 1953, so that the list of references quoted here is not intended to be exhaustive.

Water treatment

Demineralisation, *i.e.* the substitution, by suitable exchangers, of hydrogen ion and hydroxyl ion for unwanted ions in water, has continued to arouse interest, particularly in the treatment of boiler feed water. For treating water containing large quantities of calcium and magnesium as bicarbonates, carboxylic resins were found⁴ to be more economical in use than sulphonic acid resins but to require lower flow rates. Detailed operating experience has been reviewed⁵ in the light of the considerable U.S. experience of demineralisation now available.

Silica, which may be present in colloidal form, is the most difficult impurity to remove. However, highly basic quaternary ammonium-type resins such as are now available have been shown to reduce the silica content to 0.01 to 0.05 p.p.m.

For the low-cost treatment of brackish waters so as to give partial demineralisation, a process has been described⁶ which is designed to operate with optimum regenerant economy. In this process the raw water flows upward, but regeneration is performed with downward flow. The cation exchanger is regenerated with sulphuric acid to the point where the latter is just completely converted to sodium sulphate. Regenerant is thus conserved and the economics are further helped by recovery (by solar evaporation) of sodium



[Photo: Illinois Water Treatment Co.]

Fig. 1. Commercial mixed-bed demineralization unit for water treatment.

sulphate. Also, since demineralisation is incomplete, smaller volumes of purified wash water can be used without significant effect on the product. The resin is only used to about one-third of its capacity and two exchangers are used in series both for cation and anion exchangers. A preliminary softening column prevents calcium sulphate precipitation. In pilot-scale runs at the Weizmann Institute, Israel, the total solids (mainly sodium chloride) in the feed were about 2,000 p.p.m. (as CaCO_3) and the effluent contained about 300 p.p.m. (as CaCO_3). This product is considered suitable for municipal use. Cost (neglecting possible recovery of sodium sulphate) was estimated at 60 cents per 1,000 gal. treated for removal of 1,000 p.p.m. of sodium chloride. The process is considered suitable for brackish waters with not more than about 2,000 p.p.m. of dissolved solids.

Mixed-bed de-ionisation became practicable a few years ago with the production of anion exchangers with significantly lower density than cation exchangers. Regeneration is carried out by classifying the bed with upward water flow, so that the anion resin settles out above the cation resin. The cation regenerant is then in-

troduced at the boundary of the resins and withdrawn from the bottom of the column. The anion regenerant is introduced at the top of the column and withdrawn at the boundary. After regeneration the bed can be remixed with air. This system has been found⁷ more efficient than the conventional separate-bed system in respect of space requirements, regeneration time, operational time and water consumption and to give a higher resistivity product. The freedom of the mixed bed from calcium sulphate precipitation is responsible⁸ for some of these advantages. The pH of the effluent can be controlled at will by adjustment of the relative quantities of the beds. For the best results, Kunin and McGarvey recommend⁹ the use of two mixed beds in series—a weak acid/strong base combination followed by a strong acid/strong base combination. Fig. 1 illustrates a typical recent installation.

Sugar refining

Mandru,⁹ discussing the use of ion exchange in a beet-sugar factory, reported that it gave more sugar with less molasses and reduced scaling in subsequent evaporation. By comparison with phosphate treatment in the purification of thin juices, ion exchange gave¹⁰ a 13% saving in fuel consumption. Removal of nitrogenous components, as well as inorganic impurities, from sugar-beet juices has been studied.¹¹

Resin life has an important bearing on the economics of industrial ion-exchange processes and attrition loss needs to be estimated closely. This has been examined¹² with special reference to sugar-cane processing with the aid of a semi-automatic apparatus which continually cycled the resin through all the stages of its practical use and from which samples could be removed at intervals. It was found that, while there were differences in performance between different resins, cation exchangers retained their capacity much longer than anion resins. The better resins had a useful life of upwards of 1,500 cycles, which was adequate to justify the initial cost.

Allied uses examined were the preparation of lactose from cheese whey, the purification of glucose and the treatment of beers, wines, lemonade and milk.

Treatment of wastes and effluents

Ion exchange has an obvious utility in this field, which was apparently realised in Germany during the last war, since it has been stated that 17 tons/day of copper were recovered from rayon mill wastes in that country. The process is particularly suitable if the metal to be recovered occurs in, or can be thrown into, the anionic form. Thus processes have been described¹³ for recovering chromium from plating tanks (to which it is subsequently returned in the correct form and concentration), iron and chromium from pickling wastes and tin¹⁴ from tin-plate rinsing solutions. Lead has been recovered¹⁵ particularly efficiently, since its high equivalent weight makes the resin capacity high in terms of grammes of lead per ml. of resin.

A number of reviews have dealt with the use of ion exchange for removing radioactivity from effluents from, for example, atomic energy establishments. Ayres¹⁶ finds that a cation bed followed by an anion bed gives a decontamination factor of about 10^3 , which is generally adequate. Mixed-bed columns might be more efficient and cheaper.

Pharmaceuticals

For medical use or for purification of valuable pharmaceutical products, cost is relatively unimportant and these fields should give scope to ion exchangers. In the period under review the details of a process for purification of streptomycin by ion exchange have been disclosed.¹⁷ The concentration factor achieved is about 75 and the final product after further purification contains 780 units/mg., the overall recovery being about 80%. Reviews of the use of ion exchangers in pharmacy mention their application to the preparation of demineralised water. Demineralised water prepared in small-scale laboratory apparatus has been found¹⁸ not to comply fully with the requirements of the Swiss pharmacopoeia, partly because of the solubility of the resins used.

APPLICATIONS TO RESEARCH

Biochemical

Partridge¹⁹ has continued and extended his work on the separation of amino-acids from protein hydrolysates, exploiting both anion and cation exchangers. A patent application²⁰ describes the use of weak acid carboxylic resins in mixed sodium-hydrogen form. These act as buffers and the sodium-hydrogen ratio may be so adjusted that the pH of the amino-acid mixture is maintained at any selected value. Amino-acids with isoelectric points below this pH will then pass through, while those with higher isoelectric points will be absorbed. The amino-acids can thus be picked out from a mixture in order of increasing acidity. Many other workers are also active in this field which has recently been reviewed.²¹

The well-known complexes between boric acid and various monosaccharides

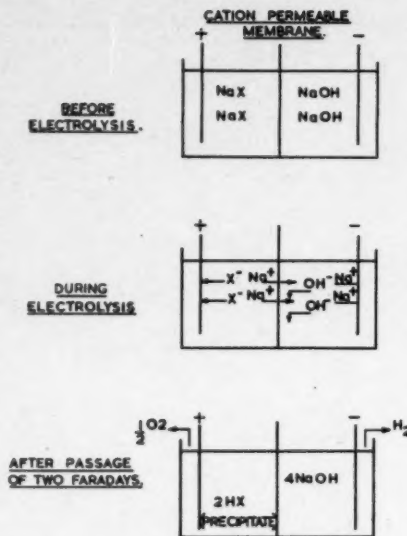


Fig. 2. Use of cation permeable membrane for preparation of sebamic acid (HX).

have been used for the separation of the latter.²² Fructose, glucose, mannose and galactose dissolved in weak sodium borate solutions were found to be adsorbed on strong-base anion exchangers. Elution with dilute sodium borate solutions and collection of the eluate in appropriate fractions gave essentially quantitative recovery of the individual sugars. Anion exchanger columns, conditioned with borax solution, have similarly been used to recover glycerol from dilute aqueous solution.²³

Inorganic separations

Work continues on the separation of rare earths by elution with citrate solutions from ion-exchange columns. Mixtures of titanium, zirconium and thorium have been resolved by the citrate-cation exchanger method and thorium has been separated from lanthanum. Hafnium has been recovered in high purity from commercial zirconium by a cation-exchange method and from an enriched hafnium-zirconium mixture by an anion exchange method. Zirconium and niobium have also been separated on anion exchangers, as have cobalt and nickel, the cobalt forming an anion complex.

The powerful technique of ion exchange combined with complex formation has now been extended to the alkali metals, by the use of uramil diacetic acid, to separate lithium and sodium from the remaining alkalis and from each other.²⁴ Ion exchangers have also been used for the preparation of organic salts of gallium²⁵ and of complex acids²⁶ such as $H_4Fe(CN)_6$ and $H_3Co(CN)_6$.

Physical chemistry

A frequent application is the use of ion-exchange resins to characterise the nature of a species in which a particular element is present. Holm, for example, has de-

scribed²⁷ a sectioned ion-exchange column, consisting of alternate bands of cation and anion resins, which can be scanned with a movable shielded Geiger-Muller counter. From the zones in which radioactivity collects it is possible to determine whether a carrier-free radioactive substance in the influent solution is present as dissolved anion, dissolved cation or in filterable suspension.

Lister and Macdonald²⁸ have used ion exchangers very widely in studying the aqueous chemistry of zirconium. They determined equilibria both on cationic and anionic exchangers under different conditions, and made estimates of ionic size from rates of uptake of zirconium on resins using different solution conditions and resins with different degrees of cross-linking. Their work suggested the existence of several anionic and cationic complexes, including polymerised species at low acidities. The polymerisation of beryllium has been studied by related techniques.

The complex-forming tendencies of copper with acetate ion, strontium and calcium with various organic liquids, and cerous ion with a variety of anions have been examined by ion-exchange methods, and the use of ion exchangers in the study of complex ions has been reviewed by Schubert.²⁹ In determining the velocity constants of the alkali-catalysed hydrolysis of amides, Widequist³⁰ has stopped the reaction after a given time by pouring the reaction mixture through a cation-exchanger column in the hydrogen form. This replaced all the sodium ions by hydrogen ions and the free acid in the eluate could then be titrated directly to determine the extent of the reaction.

Studies of catalysis

The hydrogen form of a strong acid exchanger was found to promote the hydrolysis of esters in aqueous solution more effectively than the equivalent amount of hydrochloric acid (i.e. the resin is a 'stronger' acid). Carboxylic acid resins, however, had less than a tenth of the effectiveness of sulphonated phenol types and, in acetone-water mixtures containing more than 35% acetone, hydrochloric acid was more effective³¹ than the strongest resin. Esters too large to penetrate the resin structure were shown not to be catalytically hydrolysed.³²

The inversion of sucrose has also been studied. Again the more strongly acidic resins were the more effective catalysts, but for this reaction all were less effective than hydrochloric acid. The catalytic activity was found to depend on the available surface (internal and external) of the resin and it was shown³² that diffusion of the sucrose through the resin particle was the rate-determining step. The presence of neutral salts has been shown to enhance the catalytic activity, but acid resins in other than the hydrogen form are only mildly active.

The strong-base anion exchangers have been shown to catalyse numerous other organic reactions.

Analytical applications

New uses for ion exchangers in analytical work, both organic and inorganic, have continued to appear frequently and, in so far as they have recently been reviewed by Tompkins,³³ will not be considered here in detail. One point of very general interest, however, is the use of resins for bringing insoluble materials into solution. Thus Osborn³⁴ has reported that insoluble compounds such as barium sulphate and calcium fluoride can be 'split' and thus dissolved by shaking them for some time with an excess of resin. For these materials and others more soluble the method seems to show great promise, but for less soluble compounds it is too slow.

MATERIALS

New resins

Great activity is apparent in the preparation of new resins—at least 60 references to new organic exchangers have appeared in the last two years. In addition, some work is still being done on the inorganic zeolites. The fully synthetic exchangers make up the majority of the new materials, but others which have been described are based on natural products such as coal and the bark of various trees.

A complete review is not possible, but some particularly original developments may be noted. Acidic phosphorus groups have now been incorporated into resins—both phosphonous and phosphonic cation exchangers have been prepared³⁵—and have the valuable property of selectivity for sodium over potassium. This may be particularly important for medical applications.

Guthrie³⁶ has described both cation and anion exchangers prepared by introducing active groups into cotton. The best products had capacities of 2 to 2.5 meq./g., while still retaining the physical form of the original cotton. The fabrication of belts from such materials for use in continuous processes is an obvious possibility.

Cassidy³⁷ has prepared polymers incorporating hydroquinone groups. Such materials, known as 'electron-exchange polymers,' have the property of being reversibly oxidised or reduced by ion-exchange techniques and may provide a most convenient means of performing reductions or oxidations. Another significant development is the chelating resin,³⁸ which contains groupings capable of chelate bonding with metal ions. In so far as this type of bonding involves two atoms beside the metal, steric factors exert a certain specificity and this can be further improved by suitable choice of pH.

The quaternary ammonium anion exchange resins are now sufficiently developed to be freely available as commercial products and their properties have been described³⁹ in the literature.

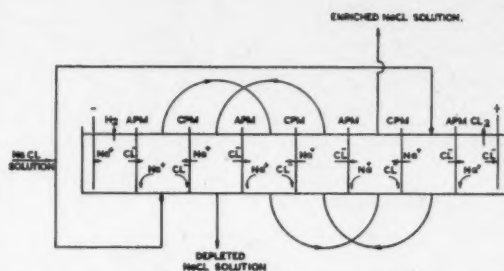


Fig. 3. Concentration of sodium chloride solution by electrolysis with ion-exchange membranes (SCH.).

APM = Anion permeable membrane.

CPM = Cation permeable membrane.

Preparation and properties of resins

The preparation of bead resins has been described by Pepper⁴⁰ and by Wheaton and Harrington,⁴¹ with particular reference to the avoidance of breakage of the beads after sulphonation.

Performance tests for the evaluation of exchangers have been described⁴² and life tests have been carried out⁴³ on several. Water-uptake and related physical properties of sulphonated polystyrene-divinyl benzene resins have been examined by Pepper⁴⁰ and co-workers, particularly as a function of the degree of cross-linking (which depends on the proportion of divinyl benzene), and by Gregor and his associates.⁴³⁻⁴⁵ Davies and Jones⁴⁶ have carried out similar measurements with a weak-base exchanger. Electro-chemical properties have also been investigated.⁴⁷

Membranes

'Permeable' membranes have been extensively studied in the U.S. and Germany in the last two years and are also available in this country. They may be used simply as diaphragms in two-compartment cells, e.g. in the electrolysis of sodium chloride,⁴⁸ where a cation-exchange diaphragm, by preventing the passage of hydroxyl ion, will prevent interaction between the chlorine and caustic soda produced at the electrodes. Another example is the preparation of sebatic acid from mono-sodium sebicate.⁴⁹ The operation of this will be clear from Fig. 2, in which R corresponds to the mono-sodium sebicate anion. More complicated arrangements are possible, and Fig. 3 indicates schematically a method⁴⁹ of concentrating a dilute brine solution by a continuous counterflow electrolytic process. Discharge of sodium and chlorine ions occurs only at the electrodes (i.e. in the end compartments) and the electrical energy is, therefore, used very efficiently. In such applications the capacity limitations of ion-exchange resins are much less relevant and it is possible to work with concentrated solutions. The considerable chemical stability of the resins can, therefore, be more fully exploited. The electrical properties of these membranes have been examined⁵⁰ by Clarke *et al.* and by Bergsma,⁵¹ who suggests their application to

electrodialysis and to the separation of sodium from potassium.

NEW TECHNIQUES

Ionic separations based upon ionic size

A sulphonated polystyrene cation exchanger is believed to consist of long hydrocarbon chains cross-linked by divinyl benzene molecules at intervals depending upon the amount of divinyl benzene incorporated (see Fig. 4). The three-dimensional cross-linked structure so obtained will have the properties

of a porous gel, the pores of which will accommodate $-SO_3O^-$ groups, the (more or less) free cations resulting from the ionic dissociation, and water molecules. For exchange to take place the exchanging ion has to find its way through these pores to an available pore or cell where it can displace the cation. Clearly, not all species capable of existing in an ionised form may be sufficiently small to penetrate the labyrinth. Thus the pores in ZK 215 (a phenol sulphonic acid resin) are larger than the effective size of the phenyl benzyl dimethyl ammonium ion, while those in ZK 225 (a sulphonated cross-linked polystyrene resin) are smaller than the effective size of the tetramethyl ammonium ion. In spite of this, quaternary ammonium ions exchange faster with the ammonium form of ZK 225 than with that of ZK 215, but the former has now a lower capacity. This suggests that larger pores occur on the outside of the particles.

It is possible, therefore, to bring about separations based upon the different rates of exchange, as was shown some time ago by Kressman and Kitchener. More recent work by Kressman⁵² has indicated that the effect is enhanced in ZK 225 by the limited capacity for the larger ions. Partridge⁵³ has used a similar type of resin, cross-linked with 10 to 12% divinyl benzene, to separate amino acids and lower peptides from proteins.

Richardson has obtained⁵⁴ particularly interesting results with the weak-base exchangers in the purification of dyes such as sky blue FF. After its metallic ions have been removed on a cationic exchanger, the dye solution, now in the acid form, is passed through the *Deacidite B* column. The smaller acid radicles such as the dye-coupling components are adsorbed, while the large dye anions are 'sieved out' on the resin structure and continue down the column. In this way pure salt-free dyes may be obtained.

Kressman⁵² has examined the possibility of using the weakly basic exchanger *Deacidite E* and also *Decolourite*, a highly porous basic exchanger. The former appears suitable for separating dyes from inorganic acids and the latter for separating dyes from one another. Similar behaviour has been noted⁵⁵ by Gustavson and Holm,

who have separated large chromium anion complexes from simpler species by using highly-cross-linked resins.

Ion exclusion

This phenomenon is superficially similar to the molecular sieve effect, but has a different origin. For the hydrogen form of a sulphonated polystyrene resin, the 'cells' which have been imagined above will have a high ionic concentration even in the absence of an external electrolyte, owing to the presence of the dissociated sulphonic groups. If the resin is now brought into contact with a dilute solution of hydrochloric acid, which may be considered as completely ionised, then the H^+ and Cl^- ions will be kept out of the cells by the high concentration of H^+ and $-SO_3^-$ ions already present. Therefore, the concentration of HCl in the liquid pores between the resin particles will be higher than that in the pores within the particles. The 'fabric' of the particle is in fact behaving as a Donnan membrane. Now, an un-ionised molecule will, to a first approximation, not be affected in this way and the 'internal' and 'external' pores will be equally accessible to it. Hence if a mixture of, say, hydrochloric acid and acetic acid is poured through the column, hydrochloric acid would be expected to 'break through' when one 'external pore volume' has passed, but the acetic acid should not break through until one 'external pore volume' plus one 'internal pore volume' have passed. Similarly, once the resin has become 'saturated' with both species, it may be rinsed with water, when the hydrochloric acid will be eluted before the more deeply held acetic acid. Thus a semi-continuous process becomes possible in which the mixture and rinsing agent are fed in alternate 'shots' to the column and the components are taken off alternately. Since the 'internal' pore volume and the 'external' pore volume are normally each of the order of 30 to 40% of the total bed volume, quite high 'capacities' are available. Moreover, since the species diffusing into the internal pores are substantially neutral, the process is much more rapid than conventional ion exchange. Separations so far achieved⁶⁶ include, in addition to hydrochloric acid from acetic acid on the hydrogen form of *Dowex 50*, sodium chloride from various alcohols, amines and formaldehyde, on the sodium form of *Dowex 50*.

Ionophoresis

Another new technique, as yet still only⁵⁷⁻⁵⁸ in the laboratory stage, is electromigration on ion-exchange resins. If, for example, a column of a cation-exchange resin is laid on its side and flushed with distilled water, while a current is passed along the column and a salt mixture is introduced near one end, then the current will be carried only by the cations and, if more than one cation is present, then a separation will occur depending on their

respective ionic mobilities. The electrodes require to be chosen so that ions from them will not overtake the ions to be separated and blur the separation. This technique may well be capable of development on a larger scale, particularly for valuable materials.

Non-aqueous solvents

A preliminary exploration of hydrogen-ion-exchange reactions between a sulphonated polystyrene cation exchanger and non-aqueous solvents has been reported⁵⁹ using various amines as solutes and a range of alcohols as well as acetone, benzene and a water-acetone mixture as solvents. The results obtained were of considerable theoretical interest and would be of particular value in design work. Systems were not chosen for study with any particular application in mind, however.

Various chlorides and nitrates in acetone solution have been equilibrated with an anion-exchange resin.⁶⁰ It seems that some complete molecules transfer to the resin phase, possibly owing to complex anion formation. The positive iodine ion I^+ has been shown⁶¹ to exchange with *Amberlite IR 100* from alcoholic solutions of iodine nitrate or sulphate. Bromine nitrate may be treated similarly. Iodine in alcohol may be estimated through the reaction:



by determining either the gain in hydrogen ion or in loss of iodine from the solution.

Column operation

Temperatures of 110°C. have been used⁶² in commercial operation on a high-capacity sulphonated polystyrene resin. The resin appeared to be quite unharmed and higher flow rates were found possible. Wilks has reviewed⁶³ the experience of available materials of construction for ion-exchange systems and made recommendations. Rubber-lined piping and vessels meet many of the requirements. Growth of micro-organisms in sugar-refining ion-exchange columns has been checked by the use of a chlorinated melamine solution.⁶⁴ Another procedure recommended⁶⁵ is to wash the column after every tenth regeneration with a liquid of opposite nature to the regenerant (*e.g.* alkali if the regenerant is acid) and then regenerate again.

The problem of displacing one liquid from a column by another with minimum mixing has been considered by Hill⁶⁶ for granular charcoal columns. The simple relations obtained should apply equally to ion-exchange systems.

Continuous countercurrent processes

Selke and Bliss have described⁶⁷ a small laboratory-scale process for removing copper from dilute waste solutions and discharging it in a more concentrated solution (up to 6.8% by weight), using a continuous ion-exchange system of two columns. The

exhaustion column, in which the copper was absorbed on the resin, was operated as a fluidised bed, and the regeneration column, from which the copper is recovered, as a moving bed. Hiester, Phillips and Fields⁶⁸ have described a number of mechanical devices, including resin metering and dewatering valves and contactors, which have been developed in aid of such a system. Another scheme, in which a flexible resin is coated on a wire gauze which can be circulated continuously through exchange, regeneration and washing tanks, has been described in the patent literature.⁶⁹

THEORY

Equilibrium

The trend of recent experimental work has been towards more careful consideration of the absorption of water and neutral molecules, as well as the ion in question, and to the proper use of activity data where available. In particular, the absorption of neutral molecules has been specifically studied by several workers.^{70, 71} Glueckauf and Duncan have included much of this work in their theoretical treatment,^{72, 73} which regards an ion-exchange resin in the metal form as a very concentrated solution of a metal resinate in the water of swelling, the osmotic 'swelling' pressure of this solution being restrained at equilibrium by the cross-links of the resin. Thermodynamic deductions on the basis of this model yield results in good agreement with experimental evidence in regions where the laws of electrolytic solutions are known accurately. Deviations at higher concentrations may reflect inadequacies in the theory of concentrated solutions. A broadly similar treatment has been given by Gregor⁷⁴ and a statistical thermodynamic approach has been put forward by Sakai and colleagues.^{75, 76}

Kinetics

Most ion-exchange column design is probably still empirical or semi-empirical, although the basis for an adequate theory has existed for some years. Recently several papers have appeared on this subject, some of them addressed directly to the chemical engineer and providing practical design methods.

The essential problem is to predict the form of the concentration history, that is the curve connecting concentration with distance down the column, at any time from knowledge of equilibrium properties of the system, rate of flow, temperature and initial conditions of the inflowing solution and the resin. This curve will advance down the column and, in the general case, will change form as it does so, although it is often a permissible approximation to assume that it remains of constant shape. The solution to the problem is obtained in principle by solving the correct equation of conservation with the correct kinetic equation. With certain simplifying assumptions regarding the

kinetics, this solution is obtained as a Bessel function which can be computed by approximate methods.

Unfortunately the assumptions regarding kinetics in some earlier treatments have been untenable in that they have assumed a particularly simple chemical step to be rate-controlling. Recent experimental work⁷⁸⁻⁸⁰ has demonstrated fairly clearly that in most practical cases diffusion through the resin particles will control the speed of the process, although at very low concentrations of the order of 10^{-3} to 10^{-4} M, diffusion through a static liquid film round the particle may be comparably, or even overwhelmingly, slow.

Of the various papers quoted above which consider this, that of Hiester and Vermeulen⁷⁷ perhaps best combines practical applicability with generality of approach and many of the other proposals made reduce to special cases of their equations. Hiester and Vermeulen first assume a kinetic equation linear in concentration which, although inaccurate, permits a reasonable mathematical treatment. They then correct for the inaccuracies thus introduced by a variable coefficient in the kinetic equation. The concentration range is divided into intervals and a mean value of this coefficient for each interval is then inserted into the equation. It thus becomes possible to fit experimental data to theoretical curves of suitable dimensionless parameters. Good agreement between experimental and predicted results is claimed and publications in which these are directly compared may be expected in the near future.

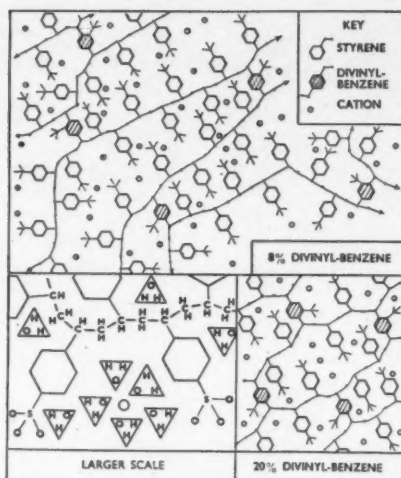
CONCLUSIONS

Considerable work has been done during the period under review, although efforts have been less in the direction of finding entirely new applications than in previous years. The tendency here has rather been for the existing applications to be consolidated and to expand slowly into neighbouring fields. On the theoretical side, a sound basis of both equilibrium and kinetic theory has been laid down and may be expected to be used to a greater degree for design.

New materials and new techniques have appeared which may change the process of ion exchange very considerably.

REFERENCES

- ¹B. A. J. Lister, *Intern. Chem. Eng.*, 1951, **32**, (8), 373.
- ²R. Kunin, *Ind. Eng. Chem.*, 1952, **44**, 79.
- ³R. Kunin and F. X. McGarvey, *Ibid.*, 1953, **45**, 83.
- ⁴F. X. McGarvey and J. Thompson, *Ibid.*, 1951, **43**, 741.
- ⁵M. B. Brines, *Power Engineering*, 1952, **56**, 82.
- ⁶K. S. Spiegler, W. Juda and M. Carron, *J. Am. Water Works Assoc.*, 1952, **44**, 80.
- ⁷A. C. Reents and F. H. Kahler, *Ind. Eng. Chem.*, 1951, **43**, 730.
- ⁸R. Kunin and F. X. McGarvey, *Ibid.*, 734.
- ⁹J. E. Mandru, *Ibid.*, 615.
- ¹⁰E. Werner, *Zucker*, 1951, **4**, 467.
- ¹¹S. Vajna and E. Pickler, *Acta. Chim. Hung.*, 1951, **1**, 196.



(Diagram: 'Endeavour')

Fig. 4. Schematic representation of cross-linked sulphonated styrene exchangers.

- ¹²J. H. Payne, H. P. Kortachak and R. F. Gill, *Jun.*, *Ind. Eng. Chem.*, 1952, **44**, 1411.
- ¹³C. F. Paulson, *Water & Sewage Works*, 1952, **99**, 199.
- ¹⁴A. B. Mindler, M. E. Gilwood and G. H. Saunders, *Ind. Eng. Chem.*, 1951, **43**, 1079.
- ¹⁵A. de Lattre, *Bull. Centre Belge Etude et Document Eau* (Liege), 1950-51, No. 7, 1526.
- ¹⁶J. A. Ayres, *Ind. Eng. Chem.*, 1951, **43**, 1526.
- ¹⁷U.S. Pat. 2,541,320 (Feb. 13, 1951).
- ¹⁸E. Butokofer and R. Ammann, *Pharm. Acta. Helv.*, 1952, **27**, 77.
- ¹⁹S. M. Partridge and R. C. Brimley, *Biochem. J.*, 1951, **48**, 313.
- ²⁰U.S. Pat. 2,549,378 (April 17, 1951).
- ²¹N. Krishnaswamy and K. V. Giri, *Science & Culture*, 1952, **17**, 369.
- ²²J. X. Khyrn and L. P. Zill, *J.A.C.S.*, 1951, **73**, 2399.
- ²³S. E. Zager and T. C. Doody, *Ind. Eng. Chem.*, 1951, **43**, 1070.
- ²⁴W. Buser, *Helv. Chim. Acta.*, 1951, **34**, 1635.
- ²⁵R. W. Blanco and J. D. Perkinson, *Jun.*, *J.A.C.S.*, 1951, **73**, 2696.
- ²⁶F. Hein and H. Lilie, *Z. Anorg. u. Allgem. Chem.*, 1952, **270**, 45.
- ²⁷L. W. Holm, *Research*, 1952, **5**, 286.
- ²⁸B. A. J. Lister and L. A. Macdonald, *J.C.S.*, 1952, 4315.
- ²⁹J. Schubert, *J. Phys. Chem.*, 1952, **56**, 113.
- ³⁰S. Widequist, *Arkiv. Kemi.*, 1952, **4**, 429.
- ³¹C. W. Davies and G. G. Thomas, *J.C.S.*, 1952, 1607.
- ³²G. Bodamer and R. Kunin, *Ind. Eng. Chem.*, 1951, **43**, 1082.
- ³³E. R. Tompkins, *Analyst*, 1952, **77**, 976.
- ³⁴G. H. Osborn, *Ibid.*, 981.
- ³⁵J. I. Bregman and Y. Murata, *J.A.C.S.*, 1952, **74**, 1868.
- ³⁶J. D. Guthrie, *Ind. Eng. Chem.*, 1952, **44**, 2187.
- ³⁷H. G. Cassidy, *Proc. Nat. Acad. Sci. U.S.*, 1952, **38**, 934.
- ³⁸R. Kunin and R. J. Myers, 'Ion Exchange Resins,' Wiley, New York, 1950, p. 63.
- ³⁹R. M. Wheaton and W. C. Bauman, *Ind. Eng. Chem.*, 1951, **43**, 1088.
- ⁴⁰K. W. Pepper, *J. Appl. Chem.*, 1951, **1**, 124.
- ⁴¹R. M. Wheaton and Harrington, *Ind. Eng. Chem.*, 1952, **44**, 1796.
- ⁴²M. Zimmermann, *Ibid.*, 1952, **64**, 107.
- ⁴³H. P. Gregor, K. M. Held and J. Bellin, *Anal. Chem.*, 1951, **23**, 620.
- ⁴⁴B. R. Sundheim, K. M. Heed and M. H. Waxman, *J. Colloid Sci.*, 1952, **7**, 511.
- ⁴⁵H. P. Gregor, F. Gutoff and J. I. Bregman, *Ibid.*, 1951, **6**, 245.
- ⁴⁶C. W. Davies and T. G. Jones, *J.C.S.*, 1951, 2615.

- ⁴⁷W. Juda, N. W. Rosenberg, J. A. Marinsky and A. A. Kasper, *J.A.C.S.*, 1952, **74**, 3736.
- ⁴⁸K. Nishida and K. Tahara, *Jap. Pat.* 178,340 (March 31, 1949).
- ⁴⁹Rohm & Haas Co., Philadelphia, Penna., 'Amberplex Ion-permeable Membranes,' 1952.
- ⁵⁰I. T. Clarke, J. A. Marinsky, W. Juda, N. W. Rosenberg, S. Alexander, *J. Phys. Chem.*, 1952, **56**, 100.
- ⁵¹F. Bergsma, *Chemisch Weekblad*, 1952, **48**, 361.
- ⁵²T. R. E. Kressman, *J. Phys. Chem.*, 1952, **56**, 118.
- ⁵³S. M. Partridge, *Nature*, 1952, **169**, 496.
- ⁵⁴R. W. Richardson, *J.C.S.*, 1951, 910.
- ⁵⁵K. H. Gustavson and L. W. Holm, *Svensk. Kem. Tid.*, 1952, **64**, 137.
- ⁵⁶R. M. Wheaton and W. C. Bauman, *Ind. Eng. Chem.*, 1953, **45**, 228.
- ⁵⁷G. Manecke, *Naturwissenschaften*, 1952, **39**, 63.
- ⁵⁸K. Spiegler and C. D. Coryell, *J. Phys. Chem.*, 1952, **56**, 106.
- ⁵⁹T. Vermeulen and E. H. Huffman, U.S. Atomic Energy Commission Unclassified Document UCRL-1989.
- ⁶⁰L. I. Katzin, E. Ceber and J. C. Sullivan, *Ibid.* AECU-1154.
- ⁶¹H. Brusset and T. Kikindai, *Chim. Anal.*, 1952, **34**, 192.
- ⁶²F. K. Lindsay, L. F. Wirth, Jun., and A. M. Durinski, *Ind. Eng. Chem.*, 1951, **43**, 1062.
- ⁶³J. F. Wilkes, 'Topics of the Month,' 1-2, *Corrosion*, 1952, **8**, (6).
- ⁶⁴H. C. Marks, U.S. Pat. 2,571, 271.
- ⁶⁵Dutch Pat. 69,718 (March 15, 1952).
- ⁶⁶S. Hill, *Chem. Eng. Sci.*, 1952, **1**, 247.
- ⁶⁷W. A. Selke and H. Bliss, *Chem. Eng. Prog.*, 1951, **47**, 529.
- ⁶⁸N. K. Hiester, R. C. Phillips and L. F. Fields, U.S. Atomic Energy Commission Unclassified Document COU-59, 1952.
- ⁶⁹S. Mihara and Y. Terasaki, *Jap. Pat.* 2223 (May 8, 1951).
- ⁷⁰C. W. Davies and G. G. Thomas, *J.C.S.*, 1951, 2624.
- ⁷¹M. S. Bhatnagar, *Ind. Eng. Chem.*, 1952, **43**, 2108.
- ⁷²E. Glueckauf, *Proc. Roy. Soc.*, 1942, **A214**, 207.
- ⁷³J. F. Duncan, *Ibid.*, 344.
- ⁷⁴H. P. Gregor, *J.A.C.S.*, 1951, **73**, 642.
- ⁷⁵W. Sakai, T. Seiyama and M. Nagamotsu, *J. Electrochem. Soc. Japan*, 1951, **19**, 343.
- ⁷⁶W. Sakai and T. Seiyama, *Mem. Faculty Eng. Kyushu Univ.*, 1952, **13**, 95-117.
- ⁷⁷N. K. Hiester and T. Vermeulen, *Chem. Eng. Prog.*, 1952, **48**, 505.
- ⁷⁸J. Grossman and A. W. Adamson, *J. Phys. Chem.*, 1952, **56**, 97.
- ⁷⁹C. N. Merriam, Jun., R. W. Southworth and H. C. Thomas, *J. Chem. Phys.*, 1952, **20**, 1842.
- ⁸⁰D. Reichenberg, *J.A.C.S.*, 1953, **75**, 589.

To Authors of Books

The Editors and Publishers of the Leonard Hill Technical Group are always ready to consider technical and scientific manuscripts with a view to publication.

All books published by us find a world-wide sale and most are reprinted regularly.

Correspondence should be addressed to:
The Chairman, Leonard Hill Limited,
Stratford House, 9 Eden St., London, N.W.1

In Future Issues

Smokeless Fuel Manufacture
New Canadian Fertiliser Plant
Ultrasonics in Industry
Chemical Engineering Reviews:
Distillation; Metallurgy

VACUUM TECHNIQUE

**Vacuum pumps and gauges; new techniques;
production of zirconium, molybdenum and titanium**

By S. L. Martin, M.Sc., A.R.C.S., D.I.C., F.R.I.C.

THIS review covers general publications dealing with high-vacuum techniques and some recent advances in pump design, vacuum gauges, general techniques and in industrial applications to vacuum metallurgy.

The journal *Vacuum*,¹ published quarterly and now in its third year, has established itself as the foremost general source of information on all fields of industry and research involving the use of low, medium or high vacua. It is less specialised than its French forerunner *Le Vide*. A particularly useful feature is the abstracts section, which is very well subdivided into groups of fairly long abstracts adequately cross-indexed and suitable for filing. Reimann² has published a new book on vacuum technique, which provides an up-to-date treatment, without complicating mathematics, of the usual sub-headings, and contains new information on seals. The papers presented at a symposium held in England on the application of freezing and drying techniques to the biochemical and biological fields have been edited by Harris.³ In a publication edited by Snyder and Lipkin⁴ on miscellaneous techniques developed on the Los Alamos nuclear project there is a chapter on 'Topics on Vacuum Techniques' in which descriptions and drawings are given of pumps, gate valves and seals, and techniques of evaporating metals. Hickman⁵ has summarised publications on high-vacuum (molecular) distillation in full, and on associated techniques in part, for the two years 1951-52. Marshall, in his general review of drying as a unit process,⁶ has similarly covered freeze drying and sublimation. The American Institute of Chemical Engineers sponsored a national meeting on vacuum engineering in May 1952, at which some 17 papers were presented, covering normal points such as types of systems, pumps, gauges, flow of gases and vapours, degassing of parts, and specialised application to low-pressure rectification, flash evaporation and aircraft engine research. According to Hickman, the attendance and display of apparatus evidenced an ever-growing interest in high-vacuum applications. It is understood that these papers will be published in *Chemical Engineering Progress*.

Vacuum pumps

Snyder and Lipkin⁴ describe in detail two new diffusion pumps of 4- and 8-in.

diameter capable of speeds of 200 and 1,200 litres/sec.⁻¹, respectively, at 10^{-6} mm. Hg pressure. George⁷ has described a simple attachment that can be made to a rotary pump for continuously freeing the pump oil from volatile solvents entrained when such a pump is used, for example, to remove petrol ether or sulphuric ether from esters of fatty acids. As indicated in Fig. 1, a small gear pump fixed on to the rotary pump cycles the oil every 4 min. to the top of a 3-ft.-long 4-in. brass pipe, sealed off 3 in. from the top; the oil flows through the row of holes externally over a steam-heated copper coil where the volatiles are evaporated, over a water-cooled coil, then into a trough for return to the pump. This device, which is said to have served for three months without the pressure in the pumped system rising above 35 microns, should be of great use in similar applications.

With oil-diffusion pumps the problem of oil contamination is ever present. Unlike mercury, the oils used may react with gases or vapours being pumped, or may decompose at the pump temperatures producing permanent gases (hydrogen, carbon monoxide, hydrocarbons) which reduce the ultimate vacua attainable or contaminate the system, despite the use of cold traps.

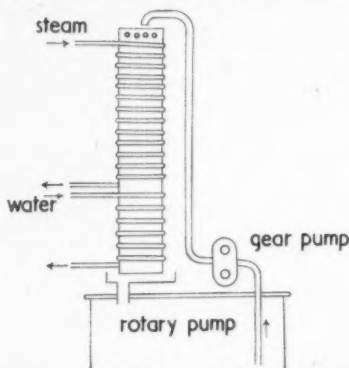
An entirely new design of pump has been described⁸—the *Speedivac F203*, illustrated in Fig. 2. This is a metal three-stage thin-film fractionation pump which is claimed to provide constant and automatic cleaning of the pump fluid from unwanted contaminants with high vapour pressure, and to be capable of maintaining

a pressure of 5×10^{-7} mm. Hg on a backing pressure of 0.05 mm. Hg. Latham and co-workers⁹ have made an assessment of various fluids used in diffusion pumps, largely on a basis of tests with this type. Their results suggest that, where the hot pump fluids are likely to be frequently opened to relatively high pressures of air, the silicone oils and chlorinated diphenyls last longer than hydrocarbon and higher ester oils. The authors also review the types of applications of mercury and of oil-diffusion pumps. After discussing cases where a decision is difficult and the factors affecting a choice, they conclude that ultimate vacuum, ruggedness and cost are generally the most important variables, the first only in operations—relatively few industrially—requiring ultimate pressures less than 10^{-6} mm. Hg. The silicones are outstanding in ruggedness. For most purposes, differences in pumping speeds, backstreaming rates and backing pressures will be insignificant for the normal oils used in fractionating pumps. The low maintenance cost of oil pumps is seen as a reason for their increasing replacement of mercury pumps, except for gas analysis and high backing-pressure applications.

Vacuum gauges

The McLeod gauge remains the only readily available absolute reference gauge, although attempts are still being made to commercialise on a wider scale the Knudsen type of absolute manometer. Steckelmacher¹⁰ has reviewed the theory and developments of glass and metal Knudsen gauges which, depending on a torque effect produced by hot gas molecules impinging on one side of a suspended vane, are independent of the nature of the gas. Although there have been many advances in design, such gauges are still, however, too sensitive in construction for other than specialised application. This is further emphasised by the work of Litting and Taylor,¹¹ who designed an automatically controlled gauge operating (on the principle of balancing a pressure-dependent radiometric force on a moving-coil instrument by an electrostatic force) over a working range of 10 to 10^{-8} mm. Hg obtained through a range change switch.

Apart from the use of low-vapour-pressure oils instead of mercury for certain applications and of Bi-Cd-Pb-Sn alloys with density 9.6 and melting at 70°C . for high-temperature applications,¹² there



[Courtesy: J. Scientific Instruments]
Fig. 1. Attachment to rotary pump for continuously cleaning pump oil (George⁷).

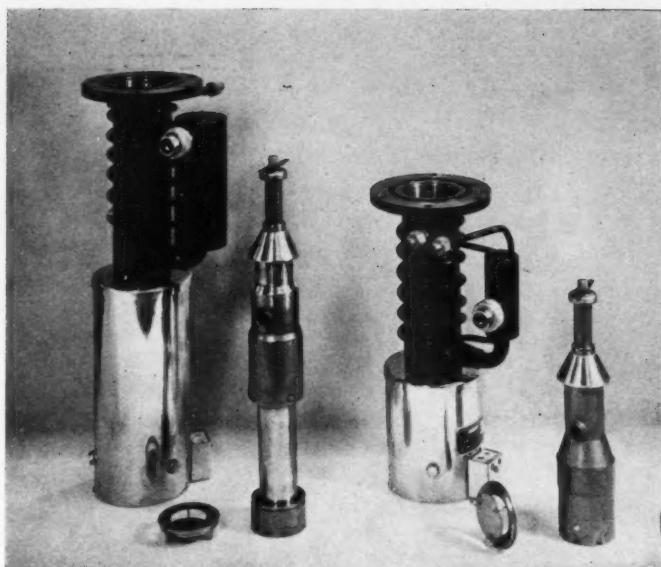


Fig. 2. New metal oil diffusion pumps. Left: fractionating type. Right: non-fractionating type (Latham).

[Courtesy: Vacuum]

has been little suggestion of change in the standard McLeod gauge form. For industrial applications the rotary form of the gauge is generally more useful—primarily as a pressure-indicating device. Axelbank¹³ has given a new design (coupled with a new type of rotary ring seal) which is portable and rugged despite the glass construction, and covers the range 10^{-2} to 10^{-6} mm. Hg. The head, shown in Fig. 3, comprises open and closed capillaries, a coil or helix, an end-reservoir, a cut-off and a rotary seal. The volumes of the parts decrease in the order V_4, V_1, V_3 , and $V_2 + V_3, V_1, V_2$. At rest, the 300-g. charge of Hg is in the lower part of the first turn. On rotation, the Hg cuts off the gas in the coil and head from the system, compresses it into the head, flows into the spillway at *A* towards *B* where it cuts off the upper end of the reference capillary, then fills V_3 and cuts off the capillary at *C* before opening the lower end (since $V_1 > V_3$). The lower end of the reference capillary is cut off at *D*, a volume of gas at the then existing pressure being trapped therein. When the upper end of the reference capillary opens, the trapped air is released into the vacuum system. In the vertical position, the gas is compressed into the closed capillary, and the excess Hg drains through the spillway, leaving the reference column at zero level. A 15-cm. length covers the required range and the overall size is $16 \times 21 \times 21$ cm. Attention should be drawn to the new tables of values for capillary depression in tubes of mercury manometers and barometers issued by the National Physical Laboratories,¹⁴ which will be useful for accurate work in medium-vacuum ranges.

Bourdon-type and other diaphragm gauges, of value because of ruggedness in low- and medium-range vacuum applica-

tions, have been receiving some attention,^{15, 16} and a British Standard Specification has been issued on Bourdon gauges.¹⁷ The gauge described by Havens¹⁵ uses a 6-cc. syphon bellows and resistance wires as detecting elements in a rugged design of small volume requiring little attention for high accuracy, yet capable of covering the range 1 atm. to 10^{-5} mm. Hg and of being used in the noses of rockets.

The circuit of the standard *Alphatron* cold-emission ionisation gauge (utilising a radioactive *alpha*-particle emitter as ionisation source) has been modified to permit connection to a commercial recorder to obtain a recording vacuum gauge covering the range 1 micron to 10 mm. Hg in sweeps of $4\frac{1}{2}$ to 24 sec. on strip or circular chart records, with slowly or rapidly changing pressures.¹⁸ The use of a polonium on nickel source in a similar-type gauge covering the range 0.1 to 100 mm. Hg has been described²⁰; the saturation current varies linearly with pressure, and the gauge is particularly useful with corrosive gases or for estimation of the composition of binary gas mixtures with the aid of calibration curves.

The hot-cathode type of ionisation gauge is capable of many modifications in circuit and gauge design, each with particular merits,^{18, 21} but the resultant instrument tends to be a specialised piece of electronic equipment. Warmholtz²² has described a tetrode type of gauge with two grids and a thin silver ion collector on the glass wall, degassable by heating and electron bombardment, which is used in a simple and stable circuit and is capable of recording down to 10^{-8} mm. Hg. The sensitivity of the more rugged and simpler Philips cold-emission ionisation gauge (in which an external magnetic field from a permanent magnet is used to lengthen the

path of the ionising electrons) has been increased. A design covering the range 10^{-1} to 10^{-7} mm. Hg has been described.²³ The outer case, acting as cathode, is of Monel metal, the looped anode of *Nichrome* wire; with 1,800 volts D.C. between anode and cathode and a permanent magnet with $\frac{3}{4}$ -in. gap providing a field of 1,700 to 1,800 gauss, 0.6 microamp current is obtained per 1×10^{-7} mm. change in pressure.

The Pirani type of gauge^{18, 21} (which depends for its action on the variation in the resistance of a hot filament, the temperature of which depends on the gas pressure and thermal conductivity of the gas at constant filament voltage) is simpler to use than the ionisation gauge and requires a less complicated associated electrical circuit, but the generally useful range is normally only 10^{-1} to 10^{-5} mm. Hg. Most firms dealing in vacuum equipment supply industrial instruments with gauge heads either in glass or metal casings. Extension of the working range up to 30 mm. Hg is theoretically possible by varying filament size and temperature, but in practice it is difficult to obtain long-term stability. From a recent study, Leck²⁴ has made the following recommendations:

- (a) Platinum filaments at around 500°K. for reliability without great accuracy at pressures up to 100 microns, e.g. on continuously pumped equipment.
- (b) For long-period stability with reasonably linear and accurate scale
 - (i) Range 0 to 100 microns—platinum or tungsten at 400 to 450°K.;
 - (ii) Range 100 to 1,000 microns—tungsten at about 750°K.;
 - (iii) Range 1 to 5 mm. Hg—tungsten at about 750°K., with no operation for long periods in oxygen.

New techniques

Medicus²⁵ suggests two very simple gasketing techniques: the use of rubber solutions (such as patching solutions) as coatings on plain ends or ground joints prior to pressure jointing; and the use of ordinary rubber bands around the coned part of unground or badly fitting ground joints. Where organic gasketing materials are undesirable, Knudsen²⁶ has found that $\frac{1}{16}$ -in. rings cut out of indium sheet 0.007-in. thick make efficient seals between metal and other surfaces when bolted every $\frac{1}{8}$ in. around the perimeter; a disadvantage is that the resultant bonding is sufficient to destroy the ring on opening the seal. For many applications it is highly desirable to be able to remove cold traps for periodic cleaning and, with large traps, ground-glass joints are likely to prove expensive or inconvenient. A trap design has been suggested²⁷ using flanged glass surfaces gasketed with neoprene rubber, which may be lubricated with an inert grease such as *Fluorothene G*.

Fremlin²⁸ presents an attractive means for providing flexible vacuum joints of

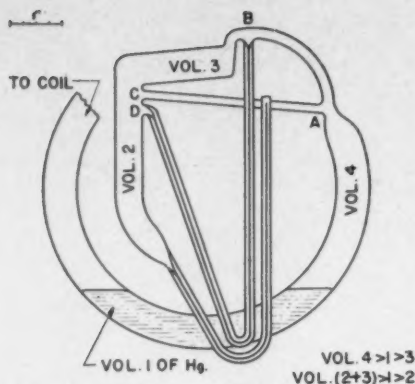


Fig. 3. New design of rotary Macleod gauge (Axelbank¹²).

large size where the bellows customarily used would be large and expensive; in addition, the joint allows better transverse and longitudinal movement and also permits rotational movement. As illustrated in Fig. 4, the joint is between flange *A* on the system and flange *B* on the moving shaft, both flanges being grooved (not shown) to take brass clamps circumferentially. *D* is a helical wire spring interlaced with flexible plastic, supporting the rubber tube *C* against collapse under large pressure differentials. Butyl rubber tube—found to be more resistant to air leakage than ordinary or neoprene rubbers—is cut from tyre inner-tubes or made from sheet by patching with rubber solution to a diameter 10% smaller than the internal diameter of the flanges, swollen by immersing in petrol, and clamped between the flanges. The leak rate of less than 0.05 litres-micron sec.⁻¹ may possibly become even less after greasing the rubber.

Greaseless stopcock

For applications where gases evolved from tap greases may contaminate the system, or where vapours which attack the greases are encountered, it is usual to use mercury cut-offs instead of vacuum taps. These are often inconvenient because of the large amounts of mercury involved, the necessity to raise and lower long lengths of mercury columns and the resultant fear of breakage. Stanier has described a greaseless vacuum stopcock which is very simple and compact and should be easy to make.²⁹ The ground surfaces are lubricated with graphite, but only mercury and glass surfaces contact the vacuum system. As seen from Fig. 5, because of the inclined axis, rotation of the plug through 180° brings the hole *A* below the mercury in the base and shuts off *B* from *C*. Lubricant is applied only to the surfaces above *D*, and the mercury in the upper half, introduced through a hole in the tap handle, feeds into the groove *D* to give a ring seal. Atmospheric pressure differentials can be sustained and a single lubrication has been found to suffice for over a year.

Vacuum metallurgy

Molybdenum. Vacuum-arc metallurgy is finding increasing applications. Parke³⁴ states that molybdenum ingots up to 1,000 lb., containing less than 0.003% oxygen, are being produced using furnaces into which the powder is continuously fed and formed into an electrode rod which is arced on to a bath of molten metal. This pure metal is finding increasing applications in high-temperature jet engines and rockets, for example.

Tantalum. Since the review two years ago,³⁰ the applications of vacuum techniques in metallurgical processes, particularly for the production of titanium,

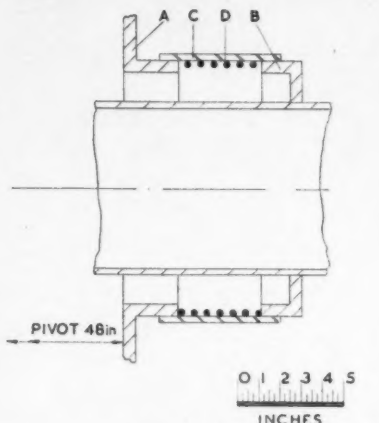


Fig. 4. Large flexible vacuum joint (Fremlin²⁵).

have increased considerably. Kroll³¹ has discussed the actual and potential uses and surveyed the performance and limitations of furnace designs, while Miller³² has described industrial-scale applications to the production of Mg, Ca, Ta and Zr. The Institute of Metals in America held a symposium on titanium in October 1952, emphasising the strategic importance of the metal.³³

A current English production method for tantalum suitable for rolling into sheets involves^{32a,b} vacuum-sintering bars pressed from powder at temperatures up to 2,500°C. by passing current through the bar in a furnace pumped by an 8-in. oil-diffusion pump backed by a rotary pump, with an overall capacity of 500 litres/sec. at 10⁻⁴ mm. Hg; hydrogen is evolved at 450 to 650°C., probably CO at about 1,500°C., and a lower oxide of tantalum at 2,000 to 2,300°C. where the ultimate vacuum is of the order of 10⁻⁴ mm. Hg. After fabrication, it may be necessary to vacuum anneal at 1,500°C.

Zirconium. In the case of zirconium,^{32a,c} the sponge metal—obtained by reducing the tetrachloride vapour with molten magnesium in an argon atmosphere—is freed from excess magnesium and magnesium chloride by heating the reduction charge in a steel retort to 950°C. in a partial vacuum of 1 to 2 mm. Hg (to lessen risk of collapse of retort) when the magnesium sublimes, and the molten chloride partly runs off and partly sublimes. Exact and cautious measurements of temperature and pressure, coupled with fast pumping, are necessary to ensure non-contamination of the highly reactive zirconium by the large amounts of water vapour evolved. The zirconium is next made molten in split-tube graphite resistor furnaces contained in steel vacuum vessels pumped to 0.3 micron Hg; this removes final traces of magnesium and its chloride. After fabricating the resultant metal into sheet, rod, tubing or wire, it may be necessary to vacuum anneal at about 750°C. for 30 min. at not greater than 2 microns Hg. Figs. 6 and 7 illustrate the vacuum furnaces used in the two stages.

Titanium production in America has increased from a few pounds in 1948 to 60 tons in 1950, 700 tons in 1951 and 5,000 tons for 1952 (Mesick³⁴), and it is hoped³⁵ that the 1951 prices per lb. of \$6 for forgings, bars and rods and \$15 for sheets and strips will soon be reduced to \$1.

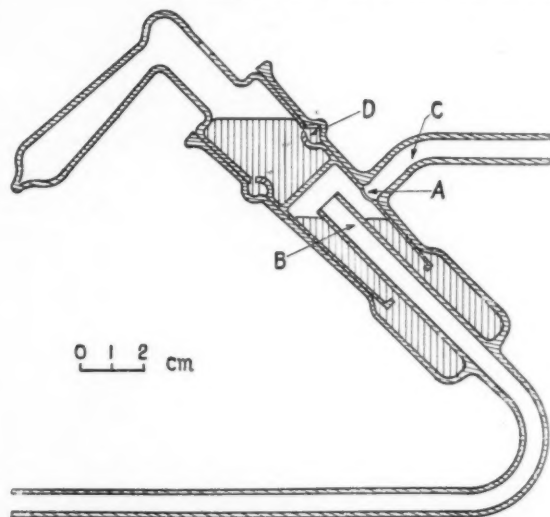


Fig. 5. Greaseless vacuum tap (Stanier²⁹).

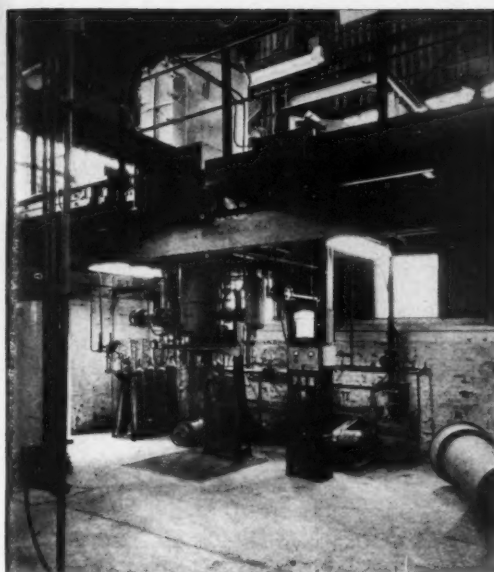
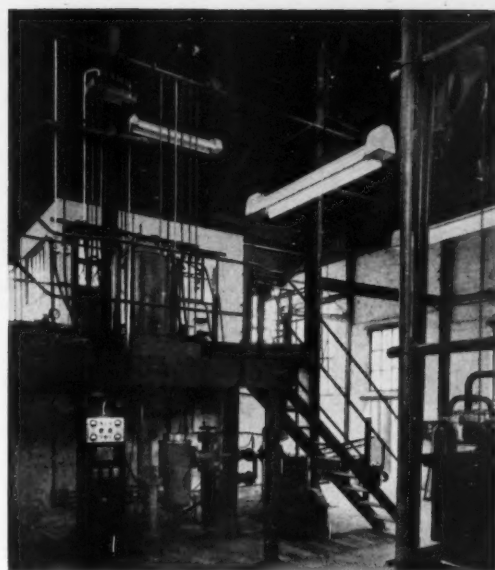


Fig. 6. Left: Vacuum furnace for eliminating $MgCl_2$ from reduced zirconium (Miller²²).

Both illustrations by courtesy of Vacuum

Fig. 7. Right: Vacuum furnace for melting zirconium sponge (Miller²²).



It has been suggested that, apart from atomic energy, there is perhaps no other industry engaged in an equal volume of simultaneous research, development and production carried out by producers, government departments and users, actual and potential.³³

Dick³³ states that under the \$150-million government sponsorship the daily production of titanium sponge should increase from a present 4 tons to over 20 tons by 1954.

This sponge (which is probably produced largely by the Kroll magnesium reduction method³⁰) is melted either in graphite crucibles in vacuum induction furnaces or by a vacuum arc process which allows build-up of large ingots without the risk of contamination with carbon. One concern has been regularly melting 1,200-lb. ingots at greater than 10 tons per week, and is now increasing the size of ingots; the largest cast ingot weighed 2 tons (Findlay³²). The metal, either in pure state or as an alloy with small amounts of Al and Cr, is finding wide applications in aircraft, ordnance and naval construction, because of its high tensile strength, corrosion resistance to sea water, low skin-resistance (low heating-up by friction when used in projected missiles or jet-engined planes) and relatively low density. For example, the Boeing XB52 plane uses 3,000 parts, totalling 650 lb. of metal, saving 430 lb. in total weight; an 81-mm. mortar base-plate, previously made of 48 lb. of steel, is now made of 24 lb. of Ti; submarine snorkel tubes, superstructure and fair-water constructions have been made of the metal or its alloys.³³

Thus titanium production is well on the way to becoming the first large-scale metallurgical process truly and permanently dependent on vacuum processes, which was not really true of the war-time process for magnesium.

REFERENCES

- ¹ Vacuum, W. Edwards & Co. Ltd., London.
- ² Reimann, 'Vacuum Technique,' Chapman & Hall, London, 1952.
- ³ Harris, 'Freezing and Drying,' Hafner, New York, 1952.
- ⁴ Snyder and Lipkin, 'Miscellaneous Physical and Chemical Techniques (Los Alamos Project),' MacGraw-Hill, New York, 1952.
- ⁵ Hickman, *Ind. Eng. Chem.*, 1953, **45**, 44.
- ⁶ Marshall, *Ibid.*, 47.
- ⁷ George, *J. Sci. Instrum.*, 1952, **29**, 413.
- ⁸ Latham, Power and Dennis, *Vacuum*, 1951, **1**, 97.
- ⁹ *Idem*, *Ibid.*, 1952, **2**, 33.
- ¹⁰ Steckelmacher, *Ibid.*, 1951, **1**, 266.
- ¹¹ Litting and Taylor, *Proc. Inst. Elec. Engrs.*, 1952, **99**, 241.
- ¹² Groszkowski, *Nature*, 1949, **164**, 886.
- ¹³ Axelbank, *Rev. Sci. Instrum.*, 1950, **21**, 511.
- ¹⁴ Gould and Vickers, *J. Sci. Instrum.*, 1952, **29**, 85.
- ¹⁵ Havens, Koll and LaGow, *Rev. Sci. Instrum.*, 1950, **21**, 596.
- ¹⁶ Dibelier et al., *J. Res. Natl. Bur. Standards*, 1951, **46**, 15.
- ¹⁷ British Standard Specification No. 1780, 1951.
- ¹⁸ Steckelmacher, *J. Sci. Instrum.*, 1951, Supp. I, p. 10.
- ¹⁹ *Rev. Sci. Instrum.*, 1950, **21**, 195.
- ²⁰ Gimenez and Labeyrie, *J. Phys. Radium*, 1952, **12**, 64A.
- ²¹ Dushman, 'Scientific Foundations of High Vacuum Technique,' Wiley, New York, 1949, p. 332.
- ²² Warmholtz and Bouwmeester, *App. Sci. Res.*, 1952, **2**, 273.
- ²³ Evans and Burmaster, *Proc. Inst. Radio Engrs.*, 1950, **38**, 651.
- ²⁴ Leck, *J. Sci. Instrum.*, 1952, **29**, 258.
- ²⁵ Medicus, *Rev. Sci. Instrum.*, 1952, **23**, 646.
- ²⁶ Knudsen, *Ibid.*, 566.
- ²⁷ Evans and Babelay, *Ibid.*, 249.
- ²⁸ Fremlin, *J. Sci. Instrum.*, 1952, **29**, 267.
- ²⁹ Stanier, *Ibid.*, 165.
- ³⁰ Martin, *INTERNATIONAL CHEMICAL ENGINEERING*, 1950, **31**, 314.
- ³¹ Kroll, *Vacuum*, 1951, **1**, 163.
- ³² Miller, (a) *Ibid.*, 1952, **2**, 29; (b) *Murex Review*, 1949, **1**, (5), 2; (c) *Ibid.*, 1951, **1**, (8), 3.
- ³³ Symposium on titanium, *J. Inst. Metals*, 1953, **5**, 131.
- ³⁴ Parke, *Metal. Progr.*, 1951, **60**, 81.
- ³⁵ O'Connor, *Chem. Engr.*, 1951, **58**, 136.

New British titanium project

The Ministry of Materials has concluded a contract with Imperial Chemical Industries Ltd. under which the company will at its own expense erect capacity to produce 1,500 tons of sponge titanium a year. The company will also install capacity for converting this metal to ingot form, and it expects production to begin in two years' time. The Ministry has undertaken to buy directly up to three-quarters of the company's output of sponge titanium over the first four years of production if the metal is not otherwise sold in various forms for use by Government contractors, and the Ministry has an option on the full output if needed for defence purposes. The price of the metal will be equivalent to the world price current at the time of purchase.

Apart from this project, the Ministry understands that I.C.I. already has in course of erection pilot plants which will begin to produce at the rate of 150 tons p.a. early in 1954.

Sturtevant products. When the Sturtevant Engineering Co. was founded in 1884 a new British industry was started, for at that time fan engineering, as we know it today, did not exist in this country and was only in its infancy in the U.S.A.

The company did not confine itself to fan engineering but began to design and construct crushing and grinding plant for special purposes such as the manufacture of fertilisers and the preparation of hydrated lime. Now the company has published a 60-page illustrated brochure, which is an index to the whole range of services and supplies it offers. Electrostatic precipitation, drying, vapour absorption, mechanical draught for boilers, air washers and filters, dust collecting and conveying systems, heating and ventilation are among the subjects dealt with.

BATCH VERSUS CONTINUOUS DISTILLATION-2

By S. R. M. Ellis, Ph.D., F.R.I.C., A.M.I.Chem.E., and J. T. Shelton, B.Sc.

(Chemical Engineering Department, University of Birmingham)

It is suggested in this paper that there is no one single answer to the relative merits of continuous and batch distillations. Numerous factors have to be taken into consideration and, in particular, the authors draw attention to the importance of the yield and the number of plates in the column in making any comparison between batch and continuous distillation. If for constant composition operation the number of plates in the column is large and the reflux ratio approaches the minimum reflux ratio, continuous distillation has the advantage. If, for reasons of economy or availability the number of plates is small, the advantage leans towards batch distillation. The first part of this paper appeared in our July issue, pp. 193-196. We again invite readers to express opinions on the results and conclusions.

IN the first part of this paper it was stated that the number of plates in the column and the yield must be taken into account when comparing the relative reflux requirements for batch and continuous distillation. The separation considered was the isolation of high-purity phenol from a feed composition of 80% phenol and 20% ortho cresol. The batch still operated with a varying reflux ratio and the continuous still with a constant reflux ratio.

In continuing this comparison the following feed and product compositions have been selected:

(b) Feed: 20% phenol, 80% ortho cresol. Product: 99.5% phenol.

(c) Feed: 20% phenol, 80% ortho cresol. Product: 90% phenol.

Feed: 20% Phenol, 80% Ortho Cresol. Product: 99.5% Phenol

Columns of 70, 50 and 35 theoretical plates were again selected with an assumed $\frac{Q}{F}$ value of 0.0007. The reflux ratio and yield calculations are tabulated in Tables 4, 5 and 6.

Discussion of results

The results from Tables 4, 5 and 6 are shown graphically on Figs. 6, 7 and 8 (numbering of tables and figures is consecutive with part 1).

As in the previous paper the final comparison is made between the reflux ratio for continuous distillation and the average reflux ratio for batch distillation. The

ratio $\frac{av. R_B}{R_C}$ is plotted against yield on

Fig. 9. The hold-up in the column has been considered only in so far as it directly influences the yield. The influence of hold-up on the curvature of the operating line and on the efficiency of separation has been neglected.

Fig. 9 shows that when the comparison is made with a large number of plates in the column and the operating reflux ratio

Table 4. Thirty-five theoretical plates

x_D	x_w	Yield %	S	S_m	R_m	R	Method of operation
0.995	0.18	6.1	36	31.9	23.2	58.0	Batch
0.995	0.14	28.8	36	33.3	29.8	92.4	
0.995	0.10	49.5	36	35.1	41.7	328.0	
0.995	0.06	68.4	36	37.7	69.7	—	
0.995	0.03	81.7	36	41.1	139.0	—	
0.995	0.01	89.9	36	46.3	418.0	—	
0.995	0.18	12.2	36	31.9	20.9	86.6	Continuous
0.995	0.14	34.9	36	33.3	20.9	121.0	
0.995	0.10	55.5	36	35.1	20.9	273.0	
0.995	0.06	74.5	36	37.7	20.9	—	
0.995	0.03	87.6	36	41.1	20.9	—	
0.995	0.01	96.0	36	46.3	20.9	—	

Table 5. Fifty theoretical plates

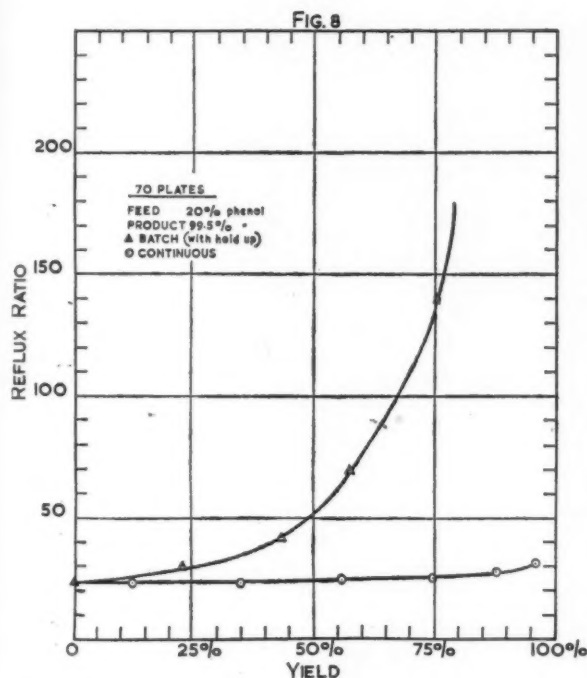
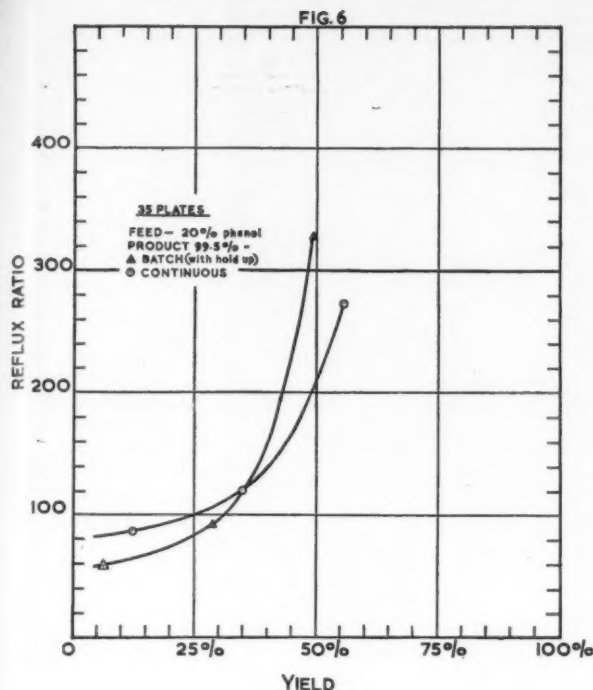
x_D	x_w	Yield %	S	S_m	R_m	R	Method of operation
0.995	0.18	3.5	51	31.9	23.2	26.5	Batch
0.995	0.14	26.2	51	33.3	29.8	33.5	
0.995	0.10	46.8	51	35.1	41.7	47.8	
0.995	0.06	65.7	51	37.7	69.7	86.3	
0.995	0.03	79.0	51	41.1	139.0	139.0*	
0.995	0.01	87.3	51	46.3	418.0	418.0*	
0.995	0.18	12.2	51	31.9	20.9	29.9	Continuous
0.995	0.14	34.9	51	33.3	20.9	31.7	
0.995	0.10	55.5	51	35.1	20.9	34.4	
0.995	0.06	74.5	51	37.7	20.9	40.3	
0.995	0.03	87.6	51	41.1	20.9	53.8	
0.995	0.01	96.0	51	46.3	20.9	103.3	

*Approximate reflux ratio

Table 6. Seventy theoretical plates

x_D	x_w	Yield %	S	S_m	R_m	R	Method of operation
0.995	0.18	0.024	71	31.9	23.2	23.2*	Batch
0.995	0.14	22.7	71	33.3	29.8	29.8*	
0.995	0.10	43.5	71	35.1	41.7	41.7*	
0.995	0.06	62.4	71	37.7	69.7	69.7*	
0.995	0.03	75.5	71	41.1	139.0	139.0*	
0.995	0.01	83.8	71	46.3	418.0	418.0*	
0.995	0.18	12.2	71	31.9	20.9	23.1	Continuous
0.995	0.14	34.9	71	33.3	20.9	23.6	
0.995	0.10	55.5	71	35.1	20.9	24.3	
0.995	0.06	74.5	71	37.7	20.9	25.4	
0.995	0.03	87.6	71	41.1	20.9	27.3	
0.995	0.01	96.0	71	46.3	20.9	31.2	

* Approximate reflux ratio



approaches the minimum reflux ratio, continuous distillation has the lowest reflux requirement. This advantage increases as the yield increases.

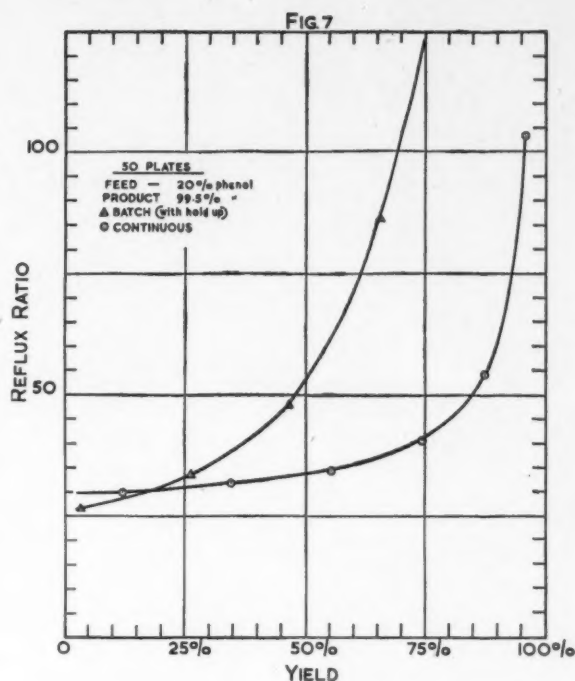
When the number of plates in the column is small and S approaches S_m , as in Table 4 and Fig. 6, batch distillation has a distinct advantage. In this instance it is not possible to obtain a yield of high-purity product much above 50%.

Comparing Figs. 4* and 9, where the same plate number and high-purity pro-

duct comparisons have been made, it appears that when the feed contains a lower percentage of the more volatile component the variation in the ratio of the reflux requirements with plate number becomes more noticeable.

From Tables 1-9* it is to be noted that for any yield of product lowest reflux requirements are obtained with a large number of plates in the column. When a comparison is made between batch and continuous distillation using reflux ratios approaching the minimum, continuous distillation has the advantage. The advantage for continuous distillation would thus appear to be easily defined. In practice, however, the economic operation of a column

is a balance between capital depreciation and operating costs. A large number of plates means increased capital costs, and high reflux ratios means high operating costs. This and other factors usually lead to the choice between batch and continuous distillation being made with a limited number of plates in the column. It may be that for a desired separation a column is available on the plant with a small number of plates. Sometimes, too, a com-



plete separation of the components is not required. Thus with a limited number of plates in the column it is seen that there is often an advantage for batch distillation.

The above comparisons involve rectifying operations giving a high-purity product and so it was thought desirable to make a further comparison under stripping conditions. Accordingly a feed composition of 20% phenol and 80% ortho cresol was selected to give an overhead production of 90% phenol. Admittedly this product composition is high for a stripping operation.

Feed: 20% Phenol, 80% Ortho Cresol. Product: 90% Phenol

The comparison has essentially been made between columns of 35, 31 and 26 plates with an assumed $\frac{Q}{F}$ value of 0.0007.

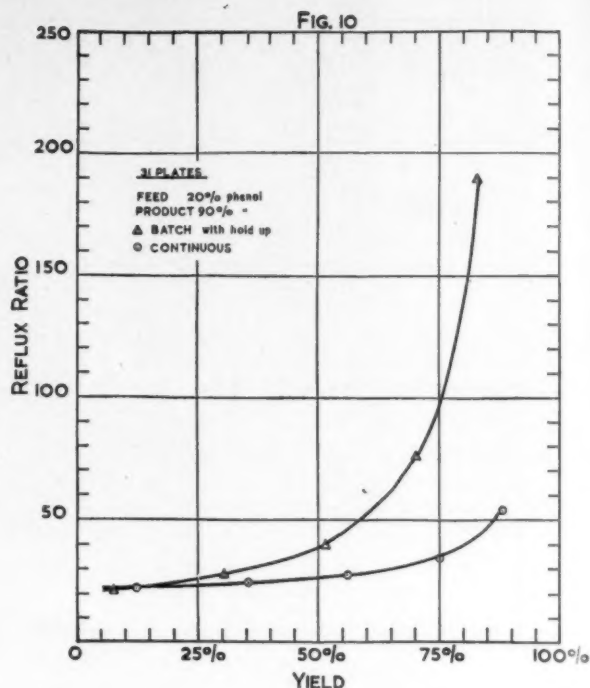
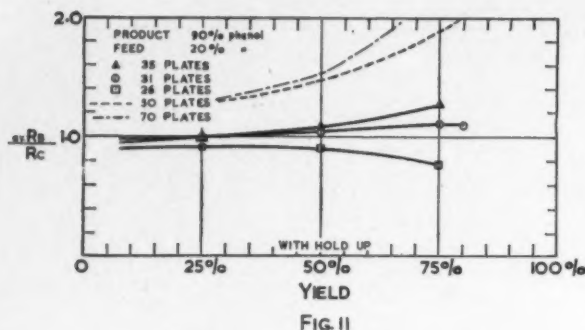
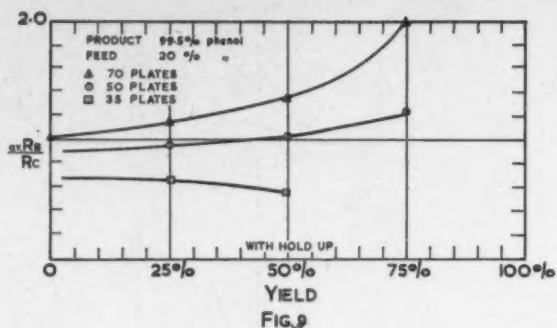
The reflux ratio and yield calculations are tabulated in Tables 7, 8 and 9.

Discussion of results

The results from Table 8 are plotted graphically in Fig. 10. Similar curves are obtained for Tables 7 and 9.

Fig. 11 shows the ratio of the average batch to continuous reflux requirements plotted against the yield of the overhead distillate. Included in Fig. 11 are also the plots for 50 and 70 theoretical plates. The conclusions are the same as previously, with a larger number of plates and high yields giving an advantage to continuous distillation.

Fig. 11 shows that a relatively small change in the number of plates in the column, i.e. 26, 31, 35, markedly influences the choice of batch or continuous distil-



lation. For a yield of 75% using 35 plates, the advantage is for continuous distillation. If a 26-plate column is available it would be advantageous to operate on a batchwise basis.

Published results

It is of interest to take the preceding calculations and see how the results compare with the conclusions of Lloyd,¹ Rose² and Perry.³

Lloyd¹ does emphasise the fact that continuous distillation gives a high yield and greater purity of product. This experimental conclusion appears to be in line with the calculations given in this paper, since Figs. 4 and 9 show that a high-purity product and a high yield means a large number of theoretical plates in the column.

It is, however, more difficult to make a comparison with the conclusions of Rose.² Firstly, Rose operates the batch column initially at total reflux before distilling at a constant reflux ratio. Thus the operating conditions are different. Secondly, he claims that the advantage for batch distillation in stripping is dependent on the amount of hold-up in the column and the yield. If the column operates with no hold-up then for a low yield batch and continuous distillation give the same results. Also the purity of the overhead product is low. As a result of our calculations it is particularly interesting to note that Rose has made the comparisons between batch and continuous distillation with a relatively small number of plates in the column. It would be extremely interesting to make further comparisons with a varying number of plates in the column

Table 7. Thirty-five theoretical plates

x_D	x_W	Yield %	S	S_m	R_m	R	Method of operation
0.9	0.18	7.0	36	17.4	20.4	20.4*	Batch
0.9	0.14	30.0	36	18.8	26.4	26.4*	
0.9	0.10	50.7	36	20.6	37.2	37.2*	
0.9	0.06	69.5	36	23.2	62.4	65.0	
0.9	0.03	82.4	36	26.6	125.0	145.0	
0.9	0.01	90.5	36	31.8	377.0	563.0	Continuous
0.9	0.18	12.5	36	17.4	18.2	21.1	
0.9	0.14	35.5	36	18.8	18.2	22.7	
0.9	0.10	56.2	36	20.6	18.2	24.5	
0.9	0.06	75.0	36	23.2	18.2	27.7	
0.9	0.03	88.0	36	26.6	18.2	38.2	
0.9	0.01	96.0	36	31.8	18.2	70.1	

* Approximate reflux ratio

Table 8. Thirty-one theoretical plates

x_D	x_W	Yield %	S	S_m	R_m	R	Method of operation
0.9	0.18	7.6	32	17.4	20.4	21.4	Batch
0.9	0.14	30.6	32	18.8	26.4	27.9	
0.9	0.10	51.4	32	20.6	37.2	39.7	
0.9	0.06	70.2	32	23.2	62.4	75.4	
0.9	0.03	83.0	32	26.6	125.0	189.0	
0.9	0.01	91.3	32	31.8	377.0	∞	Continuous
0.9	0.18	12.5	32	17.4	18.2	22.7	
0.9	0.14	35.5	32	18.8	18.2	24.4	
0.9	0.10	56.2	32	20.6	18.2	27.2	
0.9	0.06	75.0	32	23.2	18.2	33.9	
0.9	0.03	88.0	32	26.6	18.2	53.1	
0.9	0.01	96.0	32	31.8	18.2	∞	

to see if the same conclusions hold for constant reflux operations as for constant composition operation. With a larger number of plates in the column the advantage may well be in favour of continuous distillation. Of the 5% and 15% hold-up figures investigated, probably the 5% hold-up is more representative of commercial batch distillations.

The comparison given by Perry³ in favour of continuous distillation was made on the basis of a large number of plates in the column to give a high yield of high-purity product. Thus it is in line with the views expressed in this paper.

Conclusions

For rectifying and stripping operations

Table 9. Twenty-six theoretical plates

x_D	x_W	Yield %	S	Sm	Rm	R	Method of operation
0.9	0.18	8.4	27	17.4	20.4	25.0	Batch
0.9	0.14	31.4	27	18.8	26.4	33.5	
0.9	0.10	52.1	27	20.6	37.2	51.0	
0.9	0.06	71.0	27	23.2	62.4	112.0	
0.9	0.03	83.9	27	26.6	125.0	1480.0	
0.9	0.01	92.0	27	31.8	377.0	—	
0.9	0.18	12.5	27	17.4	18.2	27.2	Continuous
0.9	0.14	35.5	27	18.8	18.2	31.3	
0.9	0.10	56.2	27	20.6	18.2	39.0	
0.9	0.06	75.0	27	23.2	18.2	63.0	
0.9	0.03	88.0	27	26.6	18.2	∞	
0.9	0.01	96.0	27	31.8	18.2	—	

the relative advantage for using continuous or batch distillation depends largely on the yield and number of theoretical plates in the column. It should be remembered that this conclusion is based on a comparison using a constant reflux ratio for continuous distillation and a varying reflux ratio for batch distillation to give in each case a constant overhead composition. Also any advantage or disadvantage for hold-up in influencing the efficiency of batch distillation has been neglected.

It would be interesting to compare batch and continuous units both operating at constant reflux ratio, the comparison being made for several columns containing different numbers of plates.

REFERENCES

- ¹L. E. Lloyd, *Pet. Refiner*, 1950, **29**, 135.
- ²A. Rose, T. J. Williams and H. A. Kahn, *Ind. Eng. Chem.*, 1951, **43**, 2608.
- ³J. H. Perry, *Chemical Engineers' Handbook*, 1950, 596.
- ⁴S. R. M. Ellis, *Ind. Chemist*, June 1952, 296.
- ⁵M. J. P. Bogart, *Trans. Am. Inst. Chem. Engrs.*, 1937, **33**, 139.
- ⁶R. Edgeworth-Johnstone, *Ind. Eng. Chem.*, 1944, **36**, 1068.
- ⁷Ju Chin Chu, *Chem. Eng. Progress*, 1950, 215.
- ⁸B. F. Dodge and J. R. Huffman, *Ind. Eng. Chem.*, 1937, **29**, 1434.
- ⁹E. H. Smoker, *Trans. Am. Inst. Chem. Engrs.*, 1938, **34**, 165.
- ¹⁰E. R. Gilliland, *Ind. Eng. Chem.*, 1940, **32**, 1220.
- ¹¹A. J. V. Underwood, *Trans. Inst. Chem. Engrs.*, 1932, **10**, 131.
- ¹²M. R. Fenske, *Ind. Eng. Chem.*, 1932, **24**, 482.

Acetic Anhydride from Acetone

IMEDIATELY after the war a study of methods of producing acetic anhydride in the United Kingdom led to the conclusion that acetone, cheaply and simply produced by isopropanol dehydrogenation, would be a suitable material for anhydride synthesis. The main use of acetic anhydride is in the production of cellulose acetate, although there are minor outlets, including acetylation reactions in organic chemistry, e.g. aspirin manufacture.

With these opening comments, W. Gerard Daroux of Courtaulds Ltd. began a description of a new plant for the production of acetic anhydride from acetone at a meeting of the Chemical Engineering Group of the Society of Chemical Industry and the Institution of Chemical Engineers. His paper has now been published in the *Journal of Applied Chemistry*, 1953, **3**, 241-252.

Acetone may be thermally decomposed in a copper or silica tube according to the reaction $\text{CH}_3\text{CO}\cdot\text{CH}_3 \rightarrow \text{CH}_4 + \text{CH}_2\cdot\text{CO}$, and keten itself may decompose to ethylene and carbon monoxide thus: $2\text{CH}_2\cdot\text{CO} \rightarrow \text{C}_2\text{H}_4 + 2\text{CO}$. Other possible reactions are: dehydrogenation, and polymerisation of keten to diketene and high polymers,

sometimes with the elimination of carbon dioxide.

The main cracking reaction is slightly endothermic, to the extent of about 29,000 B.Th.U./lb.-mol., a figure which is very small compared with the sensible heat of the cracking stock, which must be heated to 1,200 to 1,500°F. It has also been shown in early experiments that the molar yield of keten diminishes with increasing conversion per pass and that at over 50% conversion the yields become uneconomic. Nickel and iron have been reported to promote the dehydrogenation of acetone.

Keten may be smoothly fixed in acetic acid to produce acetic anhydride, a reaction which evolves about 42,000 B.Th.U./lb.-mol.

The published data on the cracking process are of only slight value to the designer, and it was therefore necessary to investigate the reaction on a pilot and laboratory scale before a full-scale plant could be worked out. This work was confined largely to the cracking step, as it appeared that the absorption, recycling and recovery units could be designed by normal methods after laboratory checking of the physical data.

Full-scale plant design

Several tentative calculations for the most economical unit were involved. The variables were the conversion-selectivity relationship established by pilot work, the estimated plant cost for units having different conversions per pass, and the predicted future cost of acetone. The higher the price of acetone in relation to plant cost the lower was the conversion that could be allowed to give an economical product. The unit was eventually designed for a conversion of 25% per pass for maximum output. Lower outputs could then be obtained, if desired, with enhanced chemical efficiency.

The plant consisted of a cracking furnace, quenching unit, condenser absorbers, gas-scrubbing plant and distillation units. After the leading dimensions of the units, as fixed by the overriding economic conditions, had been decided, the main technical problems were as follows:

(a) **Furnace design.** The cracking of acetone had two special features which required more-than-usual emphasis. Firstly, the feed stock is extremely expensive, and hot acetone is a most searching material in finding leaks through jointed tubes. Secondly, the plant was to be geared to the needs of a very costly works, where any interruption of anhydride supply would be extremely expensive, if not catastrophic, so that immediate replacement of a split or faulty cracking tube was imperative.

Since available types of furnaces did not meet the requirements, the design of yet another type of furnace was undertaken. The unit consists of two identical circular cells, each designed to accommodate a 25/20 chromium-nickel welded solid-drawn coil about 400 ft. in length. Built into the walls of each cell are 24 wedge-shaped ledges which mate with lands on the eight coil supports. Each of these supports is built up from standard alloy-steel castings in three sections which are pin-jointed for flexibility. In this way the whole coil is allowed to float in the cell to avoid any stress due to thermal expansion. The first cell is used to preheat vaporised acetone to the incipient cracking temperature. The firing of the second coil is varied to give the desired degree of conversion. The cells are capped by sand-sealed refractory domed lids which support the four burners firing each cell. Dual burners are used and steam-atomised distillate oil is used for starting up the unit; when gas production has been stabilised, the oil stems are withdrawn and inspirator gas burners are used.

A high ratio of refractory area to furnace volume is of great advantage in obtaining good radiation coefficients in direct-fired furnaces. For this reason the circular cells have been divided into four sections by a battered cruciform wall which provides a good focus for flame radiation. The angle of inclination of the burners is variable, the mean position being about 5° from

vertical, facing towards the wall, so preventing flame-impingement on the tubes.

Flue gases from the cracking cell join the products of combustion in the preheating cell and the combined gases then pass to a convection section consisting of two sets of banked finned tubes, one of which is used to vaporise the feed. A second bank takes the flue-gas temperature down to 400°F. in preheating the feed to the acetone-distillation unit. The flue gases are exhausted through a hot-gas fan to the stack, and the usual by-pass flues are provided to isolate the convection section or any cell, and run on natural draught. The overall thermal efficiency of the furnace is about 85%.

(b) **Condensing and absorption equipment.** Once keten has been formed in the cracking unit it is very important to quench it as quickly as possible to prevent its thermal decomposition to ethylene and carbon monoxide. For this reason the cracked outlet gases are shock-cooled by injection of one of the recycle streams containing an excess of acetic acid. A simple four-jet unit is employed for this and an immediate volume expansion takes place on partial vaporisation of the quench feed. The pipeline is therefore considerably expanded at this point and the severe thermal strain is taken up by generously-sized expansion bellows. A single equilibrium contact is then given in a packed tower over which liquid quench is circulated, with continuous filtration to remove suspended coke blown from the cracking tubes.

Vapour at about 300°F., leaving the so-called quench tower and containing permanent gas, acetone, acetic acid, acetic anhydride and keten, is then condensed. During the condensation, combination of the keten with acetic acid takes place to form anhydride, with the evolution of heat.

The cracker products now separate into a crude liquid fraction containing the bulk of the acetic anhydride, and a gas stream containing a high concentration of acetone and some unabsorbed keten. The gas is therefore first scrubbed with a substantially acetone-free recycle stream containing acetic acid. A normal bubble-cap tower is used for this duty, but in order to keep the operating temperature low, internal recirculation through water-cooled Rosenblad exchangers is used. The effluent gas from this tower contains a little acetone, which is effectively removed by contact in a second tower through which flows the acetic acid feed to the plant. The gas is now saturated with acetic acid, which is removed by water scrubbing, and the aqueous acetic acid is returned to the acetate unit for concentration. A final caustic wash is given before the gas passes to the holder for storage as furnace fuel.

(c) **Distillation of recycle and product streams.** An acetone still produces recycle acetone from the crude product and this overhead distillate passes back to the furnace. The low conversion makes

the duty of the still relatively heavy and some thermal economy is achieved by preheating the feed in the convection section of the furnace. Also, as the feed rate is high in comparison with the overhead, the trays are split below the feed plate to accommodate the high liquid rates.

The base product from the acetone still is cooled in a Rosenblad exchanger and used as the recycle feed to the quench unit and first gas scrubber. A side stream passes forward for stripping to give an overhead of acetic acid and acetone and a bottoms product of strong anhydride and high-boiling polymers. This is distilled in a final column to give an overhead of acetic anhydride and a small residue of tarry compounds. In view of the unknown nature of these products, it was considered desirable to fit this distillation unit with duplicate re-boilers arranged with valves, so that the spare could be cleaned without interruption of production.

Spiral-plate condensers were found most suitable for all the fractionating columns; in addition to relatively low cost, they have

high heat-transfer coefficients and low pressure-drops.

The plant is almost completely automatically controlled. A central control room, slightly pressurised with fresh air, provides the focal centre for plant operation and there are few plant-variables that cannot be observed and corrected in the control room.

Operating results

As the plant has only recently begun to operate, it would be premature to discuss the long-term operating results. A remarkable sensitivity of gas make to operating temperature has been confirmed. This was shown by the pilot plant, but is much more marked on the full-scale unit. When a small change in the set point of the cracker-outlet temperature is made, the gas make changes long before the temperature change is noticed and, during normal operation, where the temperatures are usually held to within 1°F., slight ripples in the gas-make chart show minor temperature variations in the cracking coil.

Sulphur poisoning of nickel catalysts

Catalytic cracking is now an important means of producing peak load and base load water gas equivalents in the United States. This acceptance was predicated upon the use of low-sulphur feed stocks to avoid the investment costs of sulphur-purifying equipment hitherto considered necessary when using highly active nickel catalysts.

Work, described in Institute of Gas Technology Research Bulletin 10, was directed to a determination of the effects of various kinds of organic sulphur and the amounts which industrial units might be expected to tolerate when operated for the production of fuel gas. A systematic study was made of the relative performance life and activity of a commercial nickel catalyst when steam-gasifying a natural gasoline to which various sulphur compounds were added in controlled concentrations.

The results obtained in this limited laboratory system appear to have broad significance. First, the catalyst life, or capacity before regeneration becomes necessary, and the catalyst conversion efficiency, are found to vary widely and independently with the kind as well as the amount of sulphur compound present.

Second, the performance of nickel catalysts in the presence of sulphur can be improved considerably by appropriate selection of feed rates, steam concentrations and operating temperatures. Specifically, the addition of process air invariably lengthens the operating period before regeneration becomes necessary.

Third, no permanent impairment of the nickel catalyst need be caused by sulphur

in any amount or form if proper conditions of catalyst regeneration are observed. In every case it was found possible to restore the catalyst to its original effectiveness, provided excessive temperatures during regeneration were avoided. Thus the economics of sulphur purification is directly resolvable by balancing its cost against the loss of plant capacity due to down-time for catalyst regeneration and to decreased catalyst activity.

As an alternative approach, some 'sulphur-active' nickel sulphide catalysts, prepared under the auspices of the International Nickel Co. at Mellon Institute, were tested for use in catalytic cracking. Results obtained in the catalytic steam reforming of natural gas indicate that, at the relatively lower temperature levels required, nickel sulphide catalysts are not sufficiently active to be competitive with the commercial metallic nickel catalysts now in use.

Contributions and Correspondence

THE EDITOR welcomes practical articles and notes on chemical engineering and industrial chemical subjects with a view to publication. All contributions, which should be fully illustrated whenever possible, are carefully considered. A preliminary letter or synopsis is advisable. Address such material to THE EDITOR, CHEMICAL & PROCESS ENGINEERING, Stratford House, 9 Eden Street, London, N.W.1.

Letters for publication on any of the subjects covered by this Journal are also invited.

HYDRODESULPHURISATION

Shell process for removing sulphur from petroleum fractions

Hydrodesulphurisation is the name of a process developed by the Shell Group for the removal of sulphur from both straight-run and cracked petroleum fractions. This article includes a general description of the process, a simplified flow diagram for desulphurisation of a straight-run gas oil, possible applications, utility consumption figures and investment and operating costs for two 10,000 B.P.D. units charging straight-run gas oil and Middle East catalytically cracked cycle oil respectively.

THE erected costs, including contractor's overhead and profit, for hydrodesulphurisation units located in the Gulf Coast area and designed to remove 85 to 90% of the sulphur from 10,000 b.p.s.d. of straight-run gas oil (sulphur content 1.3 wt. %) and 10,000 b.p.s.d. of Middle East catalytically cracked cycle oil (sulphur content 2.8 wt. %), together with the unamortised production costs, are listed below:

	Straight-run gas oil	Middle East catalytically cracked cycle oil
Investment cost ..	\$930,000	\$1,300,000
Unamortised production cost c./bbl.	8.1	12.1

The increase in production costs for the cracked stock is due to the increased hydrogen and utility requirements. Credit has not been taken for the gasoline fraction (about 6% by weight of charge) that is produced when charging this oil.

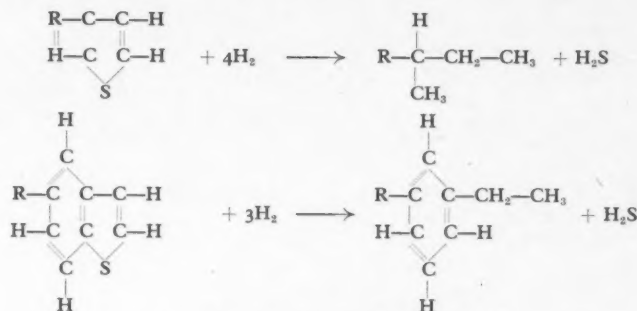
General

The Royal Dutch-Shell group have been interested in the hydrodesulphurisation field for a number of years and have carried out extensive investigations in the B.P.M. laboratories in Amsterdam, Holland. These have been directed toward the development of a more economical hydrodesulphurisation process that can be applied to a wider range of feed stocks. These studies have resulted in the development of the 'trickle process,' in which the desulphurisation is accomplished by passing the oil, in the liquid phase, together with the hydrogen, downward through a bed of cobalt-molybdenum-bauxite catalyst, or tungsten-nickel sulphide catalyst.

The development of the 'trickle phase process' has not only increased the scope of application, but the hydrogen recycle rate is reduced to a fraction of that required for vapour-phase operation. A 5,500-b.p.d. hydrodesulphurisation unit which employs this 'trickle process' is now under construction by the Lummus Co. Ltd. (to whom we are indebted for this article) for Shell refining and marketing at their Stanlow refinery. It is estimated that the investment and operating costs for this liquid-phase unit will be 80% of those required for the vapour-phase units that Shell has in operation at the present time.

Description of process

In the Shell hydrodesulphurisation pro-



Principal desulphurisation reactions.

Table 1. Feed and product properties for the trickle-hydrodesulphurisation of Middle East catalytically cracked cycle oil*

	Feed	Products†			
		Below 430° F.	430° to 500° F.	430° F. +	500° F. +
Wt. % of feed	100	9.9	20.3	87.6	67.3
Vol. % of feed	100	10.7	21.3	90.4	69.1
A.P.I.	21.3	33.8	25.4	26.1	25.5
A.S.T.M. I.B.P.-F.B.P. F. ..	446-650	270-417	437-480	475-650	532-650
Wt. % sulphur	2.90	0.01	0.02	0.34	0.43
Wt. % extractable with 98% H ₂ SO ₄	70	87	80	—	—
Octane numbers:					
Motor (clear)	—	90	77	—	—
Research (clear)	—	100 +	88	—	—
Pour pt. °F.	14	—	—	19	25
Diesel index	19.5	—	—	27.1	29.9

* Processing conditions: Co-Mo-bauxite catalyst. 750 p.s.i.a., 710° F. L.H.S.V. 1.6 vol./hr./vol. Recycle gas 1,350 S.C.F./bbl.

† Sharp fractionation.

cess, the sulphur occurring in a combined state is removed by substitution with hydrogen. Petroleum fractions, largely in the liquid state, are desulphurised by contacting with hydrogen in a fixed-catalyst bed. Two types of catalyst may be used: the cobalt-molybdenum-alumina catalyst or the tungsten-nickel-sulphide catalyst. The former is much cheaper and is desirable for desulphurisation when minimum hydrogenation of aromatics and minimum hydrogen consumption is required when treating a non-olefinic fraction containing aromatic hydrocarbons. The use of the tungsten-nickel-sulphide is applicable where upgrading of distillates by hydrogenation of aromatics is desired.

The normal operating pressures range from 300 to 750 p.s.i.a. and the average reactor temperature is approximately 700° F. For deep (80 to 90% sulphur removal) desulphurisation pressures of 600 to 750 p.s.i. are employed. Under these con-

ditions the carbon-sulphur, carbon-oxygen and, to a lesser extent, the carbon-nitrogen bonds are broken, while essentially no carbon-carbon bonds are split. If complete or nearly complete nitrogen removal from shale oils and tar sand oils is desired, the pressures and temperatures must be increased above the normal operating levels to achieve this.

The sulphur compounds in both straight-run and cracked gas oil fractions are essentially of the thiophene and benzothiophene structure with small quantities of sulphides and mercaptans possibly being present. The principal desulphurisation reactions are outlined on this page.

The resultant sulphur-free hydrocarbons from the desulphurisation of thiophenes and benzothiophenes boil at approximately 150° F. below the boiling point of the original sulphur compound. It may or may not be of advantage to remove these lighter hydrocarbons (gasoline component)

from the desulphurised oil. In the treating of Middle East straight-run gas oils only a small portion of the gasoline component is formed, while when desulphurising an oil such as Kuwait cracked cycle oil a recovery of a 400° E.P. high-octane aromatic gasoline fraction of 6% by weight of the charge oil is possible. Feed and product properties for hydrodesulphurisation of Kuwait cracked cycle oil and Kuwait straight-run gas oil are given in Tables 1 and 2.

The hydrogen consumption is dependent upon the stock that is treated and the form in which the sulphur compounds occur. Generally speaking, the consumption for a straight-run gas oil is 50 S.C.F./lb. of sulphur removed. For catalytically cracked cycle oil the consumption is approximately 65 S.C.F./lb. of sulphur removed.

The application of this process for desulphurisation is dependent upon the availability of an external source of hydrogen, but as this hydrogen need not be pure, an excellent and adequate supply may be obtained from any type of catalytic reforming operation. The effect of the diluents in the recycle hydrogen stream, namely hydrogen sulphide and methane, on the degree of desulphurisation is very slight, as shown in Tables 3 and 4. Therefore it is not necessary to remove the H₂S from the recycle stream unless it is desirable to recover the sulphur or if local ordinances prohibit the use of hydrogen sulphide containing gases for fuel.

Fig. 1 shows a flow scheme for the hydrodesulphurisation of a straight-run gas oil. The cold oil charge, together with the fresh and recycle hydrogen, is exchanged with the reactor effluent and further preheated in a furnace before being charged to the top of the reactor. The oil, which is in the liquid phase, together with the gaseous hydrogen, pass co-currently down through the catalyst bed. The effluent, after exchange with the charge, is cooled and passes to the high-pressure separator, where the remaining hydrogen is separated for recycling to the reactor. The oil is then charged to the low-pressure separator, where the major portion of the hydrogen sulphide and the small quantity of light ends formed are flashed-off. This sour gas is delivered to the plant fuel gas system. The oil is pre-heated and passes to the stripper, where dissolved H₂S and residual quantities of light ends are removed in order to meet flash-point specifications. The desulphurised oil is cooled and is delivered to the battery limits. The yield of the desulphurised oil is 100% by volume and 99 + wt. % of the charge. The characteristics for both the feed and product oils when processing Kuwait straight-run gas oil are shown in Table 2.

Commercial application

(1) **Desulphurisation of straight-run distillate.** High sulphur-content gas oils can be hydrodesulphurised into high-grade

Table 2. Hydrodesulphurisation of Kuwait gas oil by the Shell process—feed and product properties

	Feed	Product*
°A.P.I.	35.5	37.7
Aniline pt., °F.	162.5	167.5
Bromine No.	1	1
Flash P.M.C.C., °F.	253	195
Pour point, °F.	14	—
Extr. with H ₂ SO ₄ , % wt.	28	25
Conradson carbon on 10% residue, % wt.	0.02	—
A.S.T.M. distillation:		
I.B.P.	496	455
10	525	513
30	538	532
50	556	550
70	581	577
90	624	622
F.B.P.	666	662
Residue, % vol.	1.0	1.5
Loss	—	—
Hydrogen, % wt.	13.26	—
Carbon, % wt.	85.44	—
Sulphur, % wt.	1.30	0.20
Molecular weight (est.)	243	241
U.O.P., K.	11.9	12.0
Diesel index	57.7	63.1

*Properties of liquid product obtained experimentally by flashing to atmospheric pressure (i.e. without stripping out light ends).

diesel fuels, lube stocks and burning oils. In the case of desulphurisation of straight-run Kuwait gas oil (see Table 2) the increase in diesel index, along with the sulphur reduction, makes it possible to use this desulphurised oil for diesel fuel.

High sulphur-content low-value heavy naphthas can be upgraded through desulphurisation into low sulphur-content mineral spirits, and sour light distillates can be desulphurised to produce high-quality prime white distillate. These lighter compounds are more easily desulphurised than gas oils.

(2) **Improving flashed distillates as catalytic cracking feedstocks.** Hydrodesulphurisation improves flashed distillates as catalytic cracking feedstocks, for upon cracking the hydrogenated stock, a lower coke production and higher gasoline yield result, and a higher space rate is

Table 3. Effect of hydrogen sulphide in recycle gas*

L.H.S.V. vol./hr./vol. ..	2.2	2.2	1.1	1.1
Recycle gas, S.C.F./bbl. ..	270	270	1,350	1,350
H ₂ S removal from recycle gas ..	Yes	No	Yes	No
Percentage sulphur removal ..	86	86	95	94

Table 4. Effect of methane in recycle gas*

L.H.S.V. vol./hr./vol. ..	2.2	2.2
Recycle gas S.C.F./bbl. ..	1,350	1,350
Methane in feed gas, vol. % ..	0	3.8
Methane in recycle gas, vol. % ..	Negligible	15.0
H ₂ S removal from recycle gas ..	No	No
Percentage sulphur removal ..	87	84

* These data were obtained when charging Middle East light catalytically cracked cycle oil having the following characteristics and when operating at a pressure of 750 p.s.i.a. and a temperature of 708 F.:

Gravity, °A.P.I.	21
A.S.T.M. boiling range, °F.	460–640
Sulphur content, wt. %	3.1

permissible at the same total conversion. The larger the hydrogen consumption during hydrodesulphurisation, the greater are the catalytic cracking advantages. Comparing results of catalytic cracking of Kuwait flashed distillate (2.45% wt. sulphur, 0.4% wt. Conradson carbon) and the hydrodesulphurised stock (0.3% wt. sulphur, 0.08% wt. Conradson carbon with 320 S.C.F. H₂ consumption/bbl. feed) showed a 25% decrease in coke make and a 2% wt. on this charge increase in gasoline yield at a constant 50% total conversion.

As it is general knowledge that presence of nitrogen compounds impairs the activity of catalytic cracking catalysts, the resulting reduction in nitrogen content of the charge permits higher catalytic cracking catalyst activity.

The most attractive application for flashed distillate hydrogenation would be in locations containing catalytic cracking units that are short on regeneration capacity but have extra reactor capacity and ability to handle additional charge and product. Hydrogen must be available as a by-product at fuel value. This process offers the greatest advantages in the economy that places a greater premium upon high gasoline yield, increased cetane number of the catalytic gas oil, and low sulphur content. The combination of the supply of sour stocks to be refined, together with the increasing attractiveness for processes with higher gasoline yields, makes the hydrodesulphurisation of flashed distillates more attractive.

(3) **Upgrading catalytically cracked light gas oil.** Catalytically cracked gas oil can be upgraded by hydrodesulphurisation to produce a satisfactory diesel oil. The high sulphur content of Kuwait catalytic cracked light gas oil makes it unattractive except for use as a distillate fuel oil; however, upon hydrodesulphurisation, up to 6 wt. % on charge of a highly aromatic (70 to 85%) gasoline is produced with a high octane number (80 to 90 F₂ clear). A fraction distilling between 400 and 500°F. contains valuable components for power kerosene and the heavier fraction (500 to 640°F.) can be used as a diesel oil component because of its increase in diesel index and low sulphur content.

Light cycle oil can also be upgraded for recycling to catalytic cracking units. The hydrodesulphurisation of these oils results in lower coke formation, higher gasoline yield and greater ease of cracking.

(4) **Hydrodesulphurisation of de-asphalted reduced crude for catalytic cracking feed stock.** If the economics necessitate the conversion of residual fuels into distillate fuels, one possible method would be to convert flasher pitch into additional cracker feed stock by de-asphalting. For example, a typical Kuwait crude, conventionally processed, yields 32% wt. on crude of flashed distillate and 25% wt. of short residue. De-asphalting this short residue, using butane in relatively small

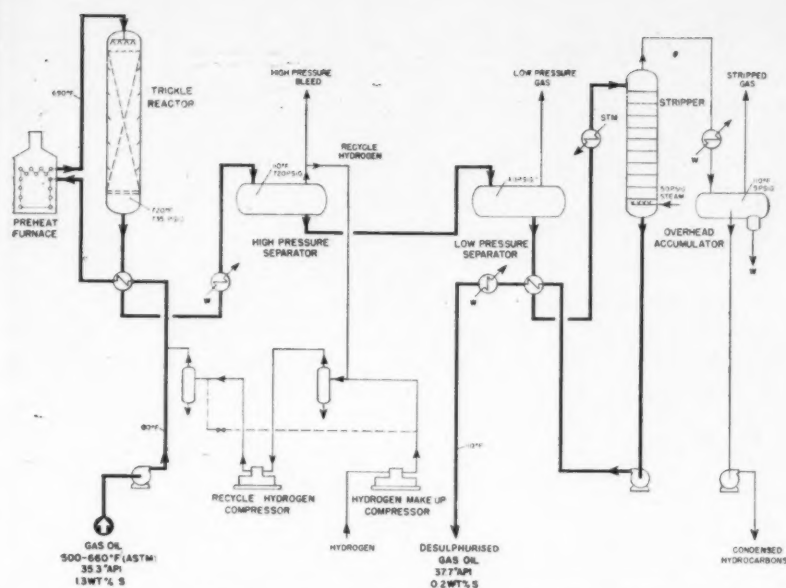


Fig. 1. Flow scheme for the hydrodesulphurisation of a straight-run gas oil.

ratios, can produce a yield of asphaltene-free oil as high as 18% wt. on crude. If this latter material is added, without further processing, to the flashed distillate feed to the catalytic cracking unit, a large increase in coke yield results. Hydrodesulphurisation of the asphaltene-free oil would decrease the coke make appreciably. Comparison of the results of catalytic cracking the hydro-treated with the untreated de-asphalted reduced crude indicates that the coke make is reduced about 35% and the gasoline yield increased about 5% on charge.

(5) **Hydrofining shale oil.** During the recent war, Germany hydrogenated Estonian shale oil and brown coal tar commercially on a large scale to produce

gasoline, diesel fuel and light lubricating oil. The B.P.M. studies have permitted considerable improvement on the German catalyst and techniques.

Colorado shale oil has been hydro-fined in the laboratory under varied reaction conditions to determine the effects of temperature, pressure and space velocity. In the usual hydrodesulphurisation range (750°F., 735 p.s.i.g. 0.54 L.H.S.V., 1,200 S.C.F. H₂ at reactor exit/bbl. feed) sulphur and oxygen were largely removed; Conradson carbon value was reduced, but the nitrogen was only partially hydrogenated. Raising the temperature to 800°F. and the pressure to 2,200 p.s.i.g. permitted all contaminants to be removed to the extent of 70 to 90%; however, the quantity of

products boiling under 700°F. did not exceed 50 vol. %, and the heavier fractions were so waxy that their suitability for use as fuels was rather questionable. Maintaining the pressure at 2,200 p.s.i.g. and increasing the temperature to 850 to 890°F. to mildly crack the waxy components, produced 80 to 90 vol. % distillate in the gasoline and gas oil range. The yields from hydrocracking at 885°F., 2,200 p.s.i.g., 0.54 L.H.S.V. and 1,200 S.C.F. H/bbl. feed were (expressed as weight %) 7% gas, 26% gasoline (40 F-2 octane number), 51% diesel oil (52 diesel index). The remaining 23% of a waxy heavy gas oil, in view of its low Conradson carbon residue (0.5%), might be suitable as catalytic cracker feed stock.

Utilities

The approximate utility requirements for processing 10,000 b.p.d. of both straight-run Kuwait gas oil and Middle East catalytically cracked cycle oil are as shown in Tables 5 and 6, respectively.

Table 5. Utility requirements for charging 10,000 B.P.D. Kuwait straight-run gas oil

Steam, lb./hr.	9,000
Cooling water, gal./min.	1,200
Electric power, kwh.	360
Fuel gas (production) M.M. B.Th.U./hr.	16
Fuel gas (consumption) M.M. B.Th.U./hr.	16

Table 6. Utility requirements for charging 10,000 B.P.D. Middle East catalytically cracked cycle oil

Cooling water, gal./min.	5,000
Electric power, kwh.	760
Fuel gas, M.M. B.Th.U./hr.	9.8

Economics

The erected cost for two units designed to process 10,000 b.p.d. of straight-run and cracked gas oil, respectively, together with unamortised production costs, are listed below. The contractor's overhead and profit is included in these cost figures and both units have been designed for 85 to 90% sulphur removal.

Rosedowns' plant. List No. 51 just published by Rose Downs & Thompson Ltd. is an illustrated pamphlet in English and French showing the latest types of equipment available for the extraction of oil from seeds and nuts, solvent extraction, refining edible and technical oils, and other purposes. Oil expeller installations, breaker rolls, expellers, a Bibby continuous rotary evaporator, deodorisers and filter presses are among the items of plant illustrated. A similar list has been published in English and Spanish.

V-belts. Simple precautions will prolong the life and increase the efficiency not only of V-belts themselves but of driving and driven machinery. The principles of V-belt care and maintenance are set out in an illustrated brochure by British Tyre & Rubber Co. Ltd.

Charge stock	Middle East straight-run gas oil	Middle East catalytically cracked cycle oil
Boiling range, °F.	496-666	446-650
Sulphur content, wt. %	1.3	2.9
Investment, \$	930,000	1,300,000
Unamortised production cost expressed as (c./bbl. charged):		
Hydrogen*	0.5	1.3
Catalyst†	1.9	3.5
Utilities‡	1.7	2.5
Direct operating labour§	2.4	2.4
Maintenance	1.1	1.6
Taxes and insurance**	0.5	0.8
Total	8.1	12.1

* At fuel value of \$0.10/M.M. B.Th.U.

† Based on catalyst life of 8,000 hr. and unit cost of \$0.80/lb.

‡ Unit costs upon which utility costs were determined are as follows:

Utility	Unit cost
Steam	\$0.40/M. lb.
Electric power	0.0065/kwh.
Cooling water	0.015/M. gal.
Fuel gas	0.10/M.M. B.Th.U.

§ Based on four men per shift at \$2.50 per hour.

|| At 4% of investment.

** At 2% of investment.

Economics of Hydrodesulphurisation

YEAST FROM MOLASSES

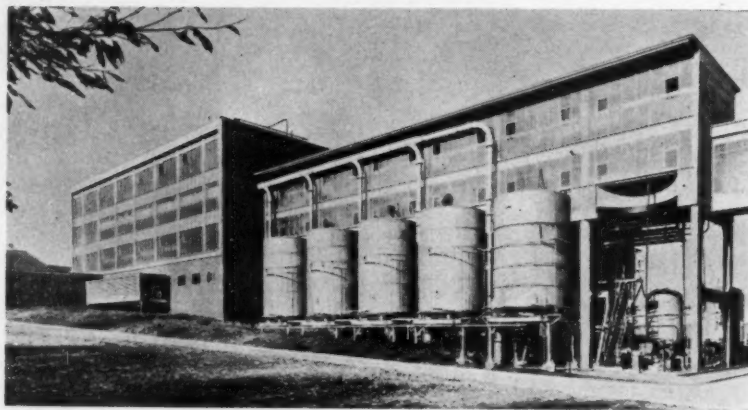
New factory engineered for automatic control and continuous processing

An outstanding example of modern 'biochemical engineering' is a new British factory for making high grade bakers' yeast from molasses at the rate of 10,000 tons per annum. Continuous processing and automatic control are used to the maximum extent and there are several other interesting engineering features, such as the location of the fermentation vessels in the open.

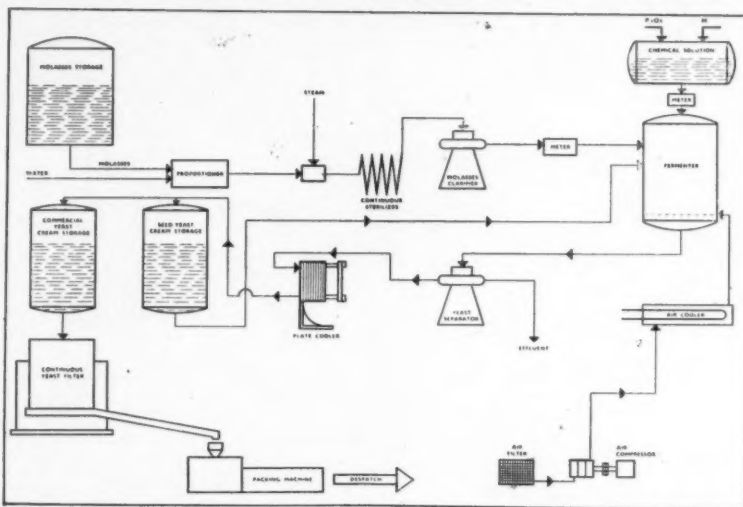
MUCH is heard today of biochemical engineering—the factory-scale utilisation of living organisms to make chemical and food products. The term largely covers fermentation, one of mankind's oldest activities. It is a useful term because it implies the application of scientific principles to an industry formerly largely an art. An excellent example of modern biochemical engineering is the manufacture of baker's yeast. For centuries yeast was the by-product of brewing and distillation. It was not until 1780 that an attempt was made to modify an alcohol fermentation to produce a better yeast. Another improvement was made in 1846 by the introduction of the Vienna method whereby the yield of yeast was increased from 4 to 6% to as much as 18% of the weight of the total raw materials.

The biggest step forward was comparatively much more recent. In 1919, a Dane, S. Sak, patented a process for making yeast entirely without the simultaneous production of alcohol. This process revolutionised the yeast industry. From being an ancillary of brewing and distillation, yeast manufacture became an industry in its own right. Gradually the raw materials which provide the nourishment for the growth of the living yeast cell have changed. Grain has been replaced by molasses and in Britain this is now the sole raw material, apart from chemicals.

Two important features of modern yeast making have been progressively improved by the application of chemical engineering principles. The first is aeration, the secret of the non-alcohol process of manufacture. By pumping large volumes of air through the fermenting wash and by adding ammonium salts at intervals during the fermentation, it is possible to obtain large yields of yeast of good quality with the simultaneous consumption, more or less complete, of any alcohol produced. The second feature is the so-called 'differential process' whereby nutrient materials are added slowly at first and then in ever-increasing amounts, at an exponential rate, as the yeast grows. This overcomes the tendency for the formation of alcohol in the presence of excess carbohydrate material and the consequent suppression of yeast growth. In short, the supply of nutrients is kept to the lowest possible rate of demand of the growing yeast.



An outstanding engineering feature of the new Standard Yeast plant is the placing of the fermentation vessels outside the building—five on each side with space for two more. The tops of the vessels are accessible from inside the control room. The building on the left houses the blowers and refrigeration plant.



Flowsheet of the yeast manufacturing process used at Dovercourt.

Some of the equipment of a modern yeast factory is similar to that needed for antibiotics manufacture. In designing the process there is the same need to safeguard against contamination by unwanted organisms which would infect the growing micro-organisms. However, in some respects the yeast manufacturer has a more difficult job than his counterpart in the antibiotics industry. The latter kills off his producing organisms when they have done their work and is only interested in the

product which can be dried and sealed against further contamination. The yeast manufacturer, on the other hand, has the problem of sending out a live organism which must be protected so that it retains its vitality until it is used by the baker. This requires the use of refrigerated vehicles.

The new factory

One of the most modern yeast factories in the world is the new plant of the Stan-



Left: Part of the separator room showing centrifuges for separating yeast cream after fermentation. Centre: Inside the fermentation plant control room where the whole process is automatically controlled. There is an instrument control panel for each fermentation vessel. Right: In the control laboratory; equipment for testing yeast by measuring the evolution of CO_2 .

dard Yeast Co. Ltd., at Dovercourt, Essex, which came into production in June 1952. This was designed and engineered by an associated company, Standard Development Co. Ltd. It produces from 5 to 10,000 tons p.a. of bakers' yeast and in addition has a considerable output of desiccated bakers' yeast which is mainly for export. Total production of yeast in the U.K. is nearly 50,000 tons p.a.

Outstanding features of this factory are:

(1) The maximum use of continuous processes, including solution and sterilisation of molasses, dehydration of the yeast cream after separation, and preparation of chemicals.

(2) The high use of instrumentation and automatic control throughout the plant. This feature is especially prominent in the fermentation control, where pH control, dosage of chemicals and anti-froth fat, etc., are fully automatic.

(3) The interesting use made of architectural design and site characteristics to rationalise production and minimise handling. For example, use is made of the natural slope of the site to place storage tanks directly underneath the buildings, so facilitating discharge of materials from road tankers and at the same time permitting maximum accessibility. Another interesting feature is the location of the fermentation vessels outside the plant buildings except for small sections of the tops of the vessels which protrude into the main control room. In this way advantage has been taken of the economy of outdoor installation (widely adopted in the oil industry) and of easy inspection and other advantages of more conventional indoor installation.

In undertaking the whole of the planning, design and process supervision of this new yeast factory, the Standard Development Co. Ltd. were fortunate in not being limited by existing buildings, and therefore had a completely free hand on the drawing board. It was possible to utilise all the company's experience of plant construction, process control and raw materials to the best advantage and, to some extent, to break away from tradition

and introduce plant and machinery specially designed for the purpose.

The position of the factory is on a sloping site overlooking the Stour Estuary. All materials are delivered over a weighbridge to the first block of buildings situated just inside the gate at the highest road level. The materials are passed through the plant with the least possible handling and the finished product is loaded into refrigerated vehicles at the opposite end of the factory.

The factory buildings consist of two production blocks and one block for laboratories and administration. The main raw material—molasses—is supplied from sugar factories nearby and all water required is drawn from boreholes on the site. Effluent is discharged into the sea.

The factory was built in a time when there were considerable shortages in building material and labour. Internal finishes have therefore been kept to a 'utility' level and effort concentrated on completely enclosed plant, eliminating the need for tiled surfaces, etc.

The floor area of the two manufacturing blocks is approximately 40,000 sq. ft., allowing easily for the production of 200 tons of bakers' yeast per week as well as a considerable quantity of desiccated yeast. The site allows ample scope for future development.

Molasses processing

Molasses is used as the sole source of carbohydrate. Molasses from various sources is discharged from road tankers by gravity into a number of tanks in the foundations of the buildings. This method enables different types of molasses to be stored separately and also allows for frequent emptying and cleaning of the tanks.

From one or more of these storage tanks molasses is drawn directly to a 2½-h.p. motor-driven proportioning pump which mixes molasses and water at a steady rate of 1 to 2 tons/hr., day and night, throughout the week. At the outlet of the proportioning pump, the molasses solution is sterilised by a continuous method, with

the supply line from the proportioning pump to the molasses clarifiers acting as a sterilising vessel. After clarification the molasses solution is passed through control instruments which automatically adjust the solution to a pre-set sugar concentration. The solution then flows to feed tanks over the fermentation vessels.

The whole process of pumping from the storage tanks and supplying the sterile molasses solution to the fermentation vessels is continuous and requires no supervision (except for the molasses clarifiers). The speed of the proportioning pumps and the rate of sterilisation is automatically controlled and synchronised with the flow to the fermentation vessels.

Chemical handling

Chemicals are used in the process as the source of nitrogen and phosphate. Solid chemicals—such as ammonium sulphate, magnesium sulphate and diammonium sulphate—are passed direct from delivery vehicles by means of a sack elevator to chemical stores at the top of the building. Liquid chemicals such as ammonia liquor, sulphuric acid and anti-froth oil are discharged from road tankers to bulk storage tanks under the roadway.

From the top floor, ammonium sulphate crystals are dissolved in a continuous flow of water and automatically adjusted to a pre-set gravity, the rate of flow being adjusted to fulfil process requirements.

Fermentation

Molasses and chemical solutions flow through measuring devices to the ten fermentation vessels from small buffer tanks situated above these vessels. The rate of flow is pre-calculated and stamped on a steel tape, which operates the flow regulators.

The 10,000-gal. fermentation vessels are of stainless steel and are cooled by means of water jackets, thus leaving the interior of the vessels completely free from obstructions except for the aeration pipes at the bottom. The aeration devices and bottoms are of a special design which enables easy cleaning of the aeration device and allows

the introduction, through the bottom, of a high-pressure rotating spray. By this means it is possible to clean the fermentation vessel in a very short time and without men entering the vessel.

The air supply is introduced through the bottom of the fermentation vessels and is controlled by a simple type of valve with remote finger-tip control.

Process control

Each fermentation vessel has its own instrument panel fitted with liquid dip gauges, air and temperature recorders, thermostatic cooling water controllers, sample devices, pH controllers, automatic anti-froth oil controllers and flow controllers for molasses and chemicals. This room also contains panels with indicator lights for plant in other parts of the buildings. Only two men are on duty in the control room; they are in complete control of the process throughout the factory.

Fermentations are set day and night according to a rigid programme in such a way that power and steam loads are kept at a nearly steady level and, since all plant is run continuously, it is utilised to the maximum.

Separation and storage

After fermentation, the contents of the vessels are pumped to totally enclosed separators where separation and washing of the yeast cream take place. One batch follows immediately after the other, so that the separators are run continuously throughout the week. The separator attendant also supervises the high-speed centrifugal molasses clarifiers placed in the same room.

The yeast concentrate is discharged from the separators through plate coolers which reduce the temperature to just above freezing point. The cold 'cream' is then temporarily stored in insulated stainless-steel vessels; these, like the fermentation vessels, are placed in the open.

Yeast pressing and packing

To meet various circumstances and demands, two types of filter are installed—continuous vacuum filters and the traditional type of high-pressure plate and frame presses.

From the filter plant the yeast passes through automatic forming, cutting and wrapping machines. To lubricate the yeast during its extrusion in the packing machines, glyceryl monostearate is added as a binder. Twenty-eight 1-lb. blocks from the wrapping machines are packed in cartons which pass through a continuous sealing machine and then, by means of conveyors, to the cold store for loading into refrigerated vehicles. These take the yeast to cold stores located throughout Britain.

Steam, air and water

As electric power is purchased from the British Electricity Authority, steam is only

used in relatively small quantities for heating water and for the steam sterilising of plant where other sterilising methods are not in use.

The boilers are oil-fired and completely automatic in operation, so that no regular attendance is required.

About 15,000 cu.ft./min. of air is used in the plant. To take the fullest advantage of the features of both high-speed turbo blowers and low-speed positive displacement blowers, both types are installed. The blowers are driven by 300-h.p. electric motors, including both variable- and constant-speed types.

Power correction equipment for the smaller motors throughout the works enables the total load power factor to be maintained at unity.

Water is pumped continuously from one or more boreholes on the site and is first used for cooling purposes and then

re-used as process water for dissolving molasses and chemicals. Hot water is partly obtained from the air coolers.

Laboratories

The plant has laboratories for biological work, baking tests, routine control and development work. Samples of each batch of yeast are tested by making up a dough, placing it in a special oven and measuring the quantity of CO₂ evolved.

The Standard Development Co. Ltd. is grouped around a team of fermentation experts with 30 years' world-wide experience in the commercial production of yeast and yeast products. Services cover design, engineering, construction (by sub-contractors) and running-in of plants of any size and, if desired, supervision of operation on a consulting basis. The company has facilities for research on all matters relating to yeast and bakers' problems.

New standards

Benzole and allied products. The following British Standards have recently been published:

'Benzoles' (pure benzole, benzole—nitration, benzole—synthesis, 90's benzole, low-gravity 90's benzole, motor benzole, low-gravity benzole) (B.S. 135: 1953).

'Coal-tar naphthas' (coal-tar solvent-naphtha (96/160), low-gravity coal-tar solvent-naphtha (96/160), coal-tar solvent-naphtha (90/160), low-gravity coal-tar solvent-naphtha (90/160), coal-tar heavy-naphtha (90/190), low-gravity coal-tar heavy-naphtha (90/190), coal-tar heavy-naphtha (90/190) (unrectified), coal-tar heavy-naphtha (90/200) (unrectified)) (B.S. 479: 1953).

'Toluols' (pure toluol, low-gravity pure toluol, toluol—nitration grade, low-gravity toluol—nitration grade, 90's toluol, low-gravity 90's toluol) (B.S. 805: 1953).

The last editions of these British Standards were published in 1939. Since then, new uses, for which the materials specified in 1939 are not suitable, have been found for benzole and allied products, and these new revisions bring the specifications into line with current commercial practice. As before, the 'Standard Specifications for Benzole and Allied Products' (1950 edition), published by the National Benzole Association, has been used as a basis for the specifications and the 'Standard Methods for Testing Tar and its Products' (1950 edition), published by the Standardisation of Tar Products Test Committee, has been used as a basis for the methods of test given in the appendices.

The main features of these revisions are the inclusion of specifications for products of lower specific gravities than those previously standardised; the deletion of industrial benzole; the inclusion of a new grade of pure benzole, namely benzole-synthesis grade, of a quality similar to benzole-nitration grade but with much

lower carbon disulphide and thiophene contents; the deletion of 95's toluol, which has been found to be no longer necessary; and the deletion of the corrosive-sulphur clause from the specifications for pure benzole and coal-tar solvent-naphtha (96/160). For those products for which it has been considered necessary to limit the corrosive sulphur, an improved copper-strip test, capable of numerical interpretation, has been introduced. (Price 7s. 6d. for B.S. 135 and 6s. for B.S. 479 and B.S. 805, respectively.)

Polythene tube for general purposes—including chemical and food industry uses—is the subject of B.S. 1973: 1953 (4s.). The standard provides for three colours of polythene tube, viz. black, white and natural, each of which is obtainable in three grades of polythene distinguished by the viscosity of the material.

Compositional and physical tests are specified and dimensions, limits and test pressures for light tubes, medium tubes, heavy tubes and tubes for screwing are laid down. An appendix gives notes on the installation of polythene tube.

Sampling fats and fatty oils. The British Standards Institution have just published a revision of B.S. 627, a document which was first published in 1935. The revision (price 3s.) has become necessary in order to provide for methods for sampling oils in road and rail tanks; also a greater number of sampling instruments has been shown than in the original edition.

In addition to the foregoing the standard, as before, provides for the sampling of liquid or semi-solid oils in bulk in shore tanks or ships' tanks, oil in course of transference from one tank to another, oil or solid fat in small tanks, drums, barrels and other small packages, and loose solid fats. Methods of collection and reduction are specified.

The Gasification of Coal

THE growing demand for gas, gasoline and chemicals has called widespread attention to the approaching need for using coal as a raw material for these products. Direct gasification, as opposed to processes in which coal is coked before gasification, is one of the most important steps in obtaining such products from coal. A technical assistance mission which visited the U.S.A. recently to study this question has now issued a report* dealing with underground gasification and the gasification of mined coal. The conclusions reached are given below.

UNDERGROUND GASIFICATION

Whatever method of underground gasification is used, a stream of air or other combustion agent (air, steam, oxygen or a mixture of the three) is led from the surface to a point in the seam which is to be gasified; it circulates through the seam in contact with the coal, with which it reacts to form CO , CO_2 , H_2 , and is brought back to the surface from some other point in the seam. The main difference between the various methods lies in the way in which the gas circulates through the seam.

(1) With the stream method, the area for gasification is marked out on three sides before the test begins; two parallel galleries are connected by a third passage known as the 'fire-face,' in which the firing is begun and maintained by a flow of comburent injected through one of the parallel galleries, the resulting gas being evacuated through the other. Combustion continues in this way, creeping back between the two parallel tracks. Thus the fire-face moves along as in blasting; the parallel lines between which it moves may run horizontally or follow the slope of the seam; this method can be used both in horizontal or sloping seams.

(2) With methods using parallel boreholes, the gas stream is driven into small-diameter holes drilled parallel to the direction of the seam. As a result of combustion, the holes gradually increase in diameter.

(3) With the percolation method, no mining operations are needed in the seam before the test is made. The gas flow circulates through the coal mass either by the natural porosity of the seam, or through cracks produced in various ways, e.g.:

- (a) Injection of air or liquid under high pressure;
- (b) Electro-carbonisation by the passage of electric current.

(4) Finally, in the underground chamber method, coal cut in the normal way is stacked up in the underground chambers produced by its removal; here, without being raised to the surface, it is gasified as in a gas producer above ground.

*The Gasification of Coal, published by the Organisation for European Economic Co-operation. 6s. net.

Experimental progress

No experiments in underground gasification (with the exception of the Russian experiments, concerning which complete information is not available) have as yet given results sufficiently satisfactory for any of the methods used to be regarded as perfect or fit to be adopted as a basis for immediate application on an industrial scale; the whole subject is still in the experimental stage.

The tests carried out in the various countries have covered widely varying geological conditions and made use of many different methods, with the result that each experience has been valuable. The subject has been surveyed from many different aspects, if not all. Some of the problems involved, such as that of making the workings gas-tight, can be satisfactorily solved immediately, as recent experiments have shown. In the case of others, such as the preparation of the workings by means other than mining, current experiments have demonstrated the efficacy of several processes which now require only to be perfected and applied to a wider range of uses.

The main problem is to maintain contact between the coal and the gas flow in spite of the progressive widening of the fire gallery. The results achieved in this direction are uneven, and it is certain that the solution of this problem will depend largely on the physical conditions of the deposit. It is as yet impossible to predict what particular solution should be applied in any particular case. The very encouraging results already achieved in a few cases offer grounds for the hope that all problems will eventually be solved; there is therefore every justification for research to be continued.

Technical and economic possibilities

Without waiting for an industrial application to place the possibilities of underground gasification beyond all shadow of doubt, it is permissible, and indeed necessary, to enquire what technical and economic results may be expected.

It should be technically possible to extract 60 to 80% of the latent heat of coal in the form of sensible heat and latent heat of gas. In the least favourable prospect there will be the sensible heat of the gas, to be used in boiler plants or gas turbines, but it may be hoped without exaggerated optimism that at least 50% of the latent heat of the coal may be tapped in the form of latent heat of gas, i.e. in an easily transportable form. The quality of the gas will depend on the combustion agent used: producer gas mixed with air, synthesis gas mixed with steam alternating with air or in a continuous mixture with oxygen.

From the economic standpoint, it is still too soon to form any opinion. The question of whether a process will prove

a commercial proposition usually depends as much on the perfection of the technique by which the principles on which it is based are applied as on the principles themselves. Nevertheless, the reduced amount of preparatory mining required, or indeed its abolition altogether, is a very important point in favour of underground gasification, an argument which is increasingly supported by recent developments.

In any case, even if the cost per calorie of the gas obtained from underground gasification was of the same order as the corresponding cost using traditional methods, the former method would still have the great advantage of doing away with mining operations, for which labour is increasingly difficult to obtain, and of producing a gas which, if the combustion agent used is oxygen, offers a wider range of uses and is more economical in transport than the solid fuel.

The trend of future experiments

As the value of continuing experiments in underground gasification is no longer in question, it now remains to ascertain the best way of continuing them.

As previously mentioned, the main problem still to be solved is that of designing workings in such a way as to maintain contact between the coal and the gas. Analysis and experiment have demonstrated the conditions under which the methods adopted so far can be operated satisfactorily: for instance, in the stream method, the burnt-out area must form a gas-tight barrier behind the fire-face, whereas with the parallel bore-hole and unidirectional flow method, the burnt-out area must remain to some extent porous in order to allow the combustion agent free passage. A more general consideration arising from this is that the layout of the workings must be adapted to suit the behaviour of the particular strata. Quite obviously this problem can only be solved on the spot. Hence, further underground experiments will have to be made. They will have the advantage of the considerable advances, both theoretical and practical, made during the past two years.

Surface experiments with models, within the carefully defined limits of a physical and chemical study of the gasification of coal in the mass, have made a valuable contribution to the comparative study of the various combustion agents and the development of new types of layout designed to overcome the difficulties inherent in the formation of burnt-out areas which shall be permeable to the gas flow. These experiments using models have revealed several of the fundamental relations between the main factors involved in the integral gasification of coal in the mass. Many researchers believe that work should continue along these lines with a view to building on the nucleus of infor-

mation that has been accumulated so far.

It is to be hoped that future work will maintain and indeed accentuate the purely scientific character of the preceding investigations. In particular, what has already been done shows that there is room for considerable progress in the study of the kinetics of reactions. Similarly, it would be of value to obtain further knowledge concerning the transmission of heat in rock walls and roof, and to study the part played by factors relating to the source of heat and its movement, together with those which govern the thermal characteristics of the rock itself.

GASIFYING MINED COAL

Chapter 2 of the report deals with the processes of gasifying mined coal which are in use in Europe or of which the experts received practical demonstrations at a number of research centres and pilot plants in the United States. The chapter also shows how various gas producers work and describes the results achieved.

Under the American methods, the fuel is previously ground very fine, and then gasified in suspension. European methods, on the other hand, are based on the fluidised or Winkler state, and treat the fuel in a cruder state (mesh 0 to 8). This is the point on which gasification problems in America differ from those in Europe.

The main features of these problems are:

(a) **Supplying the heat required for water-gas reaction.** Three methods are available to supply the heat required for the gasification of particles in suspension: either (i) the endothermic gasifying agents (steam, circulating gas flow) are considerably superheated, or (ii) they are mixed with exothermic substances (oxygen), or (iii) simultaneously mixed with a smaller quantity of oxygen and moderately superheated steam.

The exclusive use of endothermic gasifying agents superheated to a very high temperature (steam) has been abandoned in the United States because of the very rapid wear of pebbles in pebble heaters, and also because the great quantity of gas required to heat the pebbles raises the operating costs far above the cost of using oxygen.

Pulverised coal is therefore gasified by adding Linde-Franks oxygen to the flow.

Oxygen is also used in Europe, but experiments are also being carried out in fluidisation plants with a view to producing a synthesis gas without using oxygen. In this case the reaction heat needed for the manufacture of water gas is supplied by preheating the fuel either with a blast of hot air (I.C.I.; B.A.S.F.-Flesch-Demag) or directly from heating surfaces (Fuel Research Station, London). These methods can only be used with a heavy concentration of fuel, as in fluidisation.

(b) **The removal of gasification waste.** This often causes trouble when operating above the melting point of the ash. Owing to the rapid passage of the

coal through the generator, the gasification of the fuel in suspension must take place at a relatively high temperature, unless only types of coal particularly suitable for the reaction are selected. Hence, American research must be directed towards finding a solution for all problems relating to the caking of slag, e.g. the cooling of generator walls, cooling of the gas by circulation in closed circuit, heat losses and resistance of the walls.

When gasifying coal in the fluidised state, the fuel remains longer in the generator and it is therefore possible to work at lower temperatures.

(c) **Feeding gasification fuel to the generator.** This is particularly important in the case of the gasification of pulverised coal in suspension, for in this case the generator operates with no reserve of fuel.

The amount of coal to be gasified per unit of time has to be fed into the generator continuously and at an accurately determined rate to prevent losses of fuel or the formation of explosive mixtures of gases. Measures such as substituting several feeders for one single feeder to avoid the risk of a breakdown of any one of them, or recycling part of the gas emerging from the generator in order to burn off excess oxygen, have by no means solved the problem.

These thorny problems do not arise in processes based on the fluidised state, as the gasification chamber always holds enough coal for one or two hours' running.

American and European achievements

Apart from questions of personal preference, more general considerations may explain why gasification experiments in the United States have taken a different direction from those in Europe.

Research workers in the United States are much more intent than in Europe on finding solutions applicable to all cases, and coal is usually most easily obtained in the pulverised form.

Obviously, more money and time will be needed to produce a universally applicable solution. The United States have more money and especially more time, since Europe must put gasification processes into practice immediately, whereas the United States consider them mainly as a long-term proposition.

The main purpose of American research is to discover a way of gasifying caking coals which can best be treated when they are as finely broken up as possible, i.e. precisely in the pulverised form.

Among the results achieved in the United States, special mention should be made of a way of solving the problem which is of capital importance in the gasification of pulverised coal in suspension, namely of the continuous feeding of suitable quantities of fuel into the producer. The United States Bureau of Mines fluidised feeder and the Texaco slurry feeder represent two such solutions. The former,

which seems wholly satisfactory at atmospheric pressure and promises to be fully successful at high pressures, was seen in operation by the members of the mission. There are still difficulties to be overcome in the case of the slurry feeder owing to choking and erosion in that part of the superheater where the slurry water is turned to steam.

Generally speaking, heat losses are greater in the case of gasification of pulverised coal than in fixed-bed or fluidised-bed gasification, because the gasification chamber is larger. Heat losses become particularly noticeable when all the surfaces of the reaction chamber have cooled down. Attempts are being made to counter this drawback by gasifying pulverised coal under pressure. Owing to the increased capacity of the apparatus in this case, the size of the reaction chamber is relatively smaller. The method of feeding described above is especially suitable for gasification under pressure.

It is likely that, once the economic efficiency of the plant is no longer jeopardised by the cooling of the reaction chamber walls, a better arrangement will be made for evacuating slag in molten state when gasifying pulverised coal in suspension.

Among the results of experiments in Europe, two methods may be mentioned. The first is the apparatus devised by the I.C.I. for carrying the fine product and at the same time separating two reaction chambers; the other embodies the principle underlying the B.A.S.F.-Flesch-Demag process, namely that a gas of high purity can be produced at very low temperature from very finely pulverised fuel or even from finely granulated coal.

A survey of the results achieved by gasification experiments in American and in Europe shows that, despite the different directions that research has taken, considerable progress has already been made towards the solution of the general problem of gasification. From this point of view, research on both sides of the Atlantic is seen to be wisely directed towards the attainment of a common goal: the development of a universal and economically efficient gas producer.

This goal will be attained all the sooner if the partial solutions arrived at in the United States are more rapidly brought to the notice of European research workers and *vice versa*.

Nickel and oil refineries. 'Wiggin Nickel Alloys' No. 19 contains a description of the Fawley petroleum refinery and short articles on oil refinery pumps, medical research cage units, *Santon* industrial immersion heaters, coke oven corrosion problems, improving diesel engine efficiency, recording thermometers, furnace belts and precision dispensing. Copies of this journal may be obtained by writing to Henry Wiggin & Co. Ltd., Birmingham.

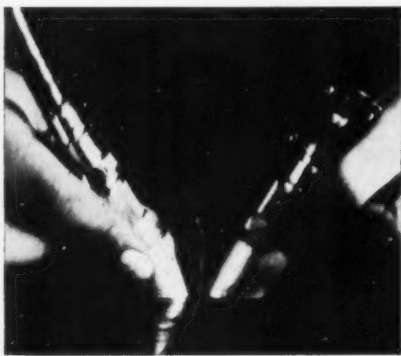
Ultrasonic Testing of Welded Chemical Plant

ULTRASONIC techniques have been available for the non-destructive testing of thick metal sections for some 20 years. These techniques have been applied in the foundry for testing ingots and large forgings for at least a decade. However, the ultrasonic method was not really satisfactory for studying welds of metals, say $\frac{1}{2}$ in. to 5 in. thick, until quite recently. One of the first major successes of the improved technique was during the construction of the Margam steel works in South Wales, when every major weld of the framework of the buildings was tested by ultrasonic inspection.

This method, developed by W. S. Atkins & Partners, is now being applied to the testing of pressure vessels, fractionating plant, heat exchangers and similar equipment by W. J. Fraser & Co. Ltd., chemical engineers.

New technique

Industrial equipment made from 1-in.-thick steel plate may suffer from two major types of fault: unsatisfactory welding and laminations or inclusions in the steel plate used in the manufacture. Apparatus for detecting cracks, incomplete fusion and similar welding faults for this type of plant



The probes with special plastic holders in position on a weld.

constructed from thick plate has not been satisfactory in the past. The chief reason has been the use of too low an ultrasonic frequency and unsuitable probes or other means for introducing the ultrasonic waves into the object being tested.

Welded plate for pressure vessels and general chemical plant can now be confidently inspected by an improved ultrasonic method using waves at 5 mc/s. and wedge-type plastic probes used in a special technique. Cracks, porosity, inclusions and laminations are found with certainty, if present, and can be checked by X-radiography if need be. Clad steel can also be checked for lack of adhesion between the

metal surfaces—a very valuable test where clad metal is used for spinning end caps.

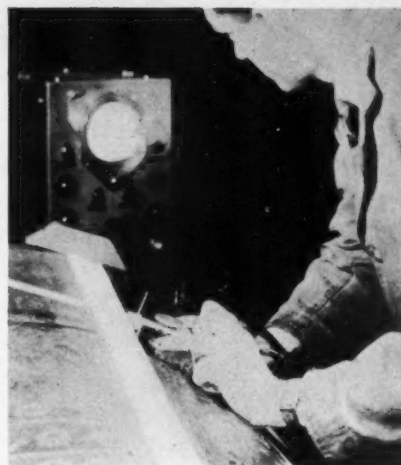
At Fraser's, welds of metal plate of the order of 1-in. thickness are inspected ultrasonically by projecting the sonic beam probe towards the weld from a distance a few inches away and testing for any reflected waves by means of a record receiver probe close beside the injector or transmitter. Any air-metal interface is an excellent reflector of these high-frequency sound waves. If the receiver probe detects an echo or reflection there is a fault present. Its position in depth, area and probable nature can be found by moving the probe positions and checking by radiography if necessary. The length of a weld is relatively rapidly checked by running the probes along parallel with the weld.

Before using the ultrasonic test little preparation is necessary except the removal of loose surface scale and the application of a grease or oil to give good sonic contact between the probe and metal. In practice, each weld problem is treated on its merits as a more or less special case, thus butt and pipe welds are treated differently. The chief reason for this is that an optimum direction of propagation exists for the ultrasonic waves to give greatest sensitivity. This direction depends largely on the type of fault anticipated and its planar position with respect to the surface from which one is testing. It is extremely difficult to test stainless steel by this technique, as its grain size is unfavourable, causing very heavy scattering of the waves and hence very low sensitivity of the equipment.

Low-temperature equipment

Inclusions and laminations do not give serious trouble unless they outcrop at the welded edge. In such a case they can cause doubts about freedom from notch brittleness at temperatures from 0 to -40°C . Such low temperatures develop when fractionating columns are suddenly shut down and one end may become notch brittle if dirty steel has been used in its construction. Vessels for carbon dioxide, butane, propane and general petrochemical storage and reaction at these temperatures must be free from possible notch brittleness. Thus the steel used in their fabrication must not contain laminations or rolled-out inclusions.

An example of this occurred recently when testing a completed refinery fractionating column intended to work down to -40°C . On completion a manhole was cut into one end of the column. By chance this segment was then cut by machine for a laboratory demonstration and showed a bad case of numerous inclusions, along the cut edge, which had not been



A test in progress showing the operator using the special probes and observing the ultrasonic waves on a screen.

detected either during the standard visual inspection of the steel plate used, nor by X-radiography, which had been used on half the welded seams.

The presence of multiple rolled-out inclusions in this specimen led to doubts about the operating safety of the whole column. These suspicions were only dispelled when the whole length of every weld had been examined by the new ultrasonic technique. It was then found that no welds had been made on to inclusions and that the particular plate from which the manhole was cut was the only one which happened to be dirty steel. This was considered to be a major success for the novel technique, as not only was the cost of a new column saved but also the time delay of rebuilding it. The actual column was 4 ft. in diameter by 110 ft. long.

Another example was when a large, steam-jacketed mild-steel mixing vessel had been fabricated. Both the jacket and the body were tested by means of test plates which were part of the actual job, receiving the same stress relief *in situ* as the welded vessel. Both these plates failed when tested owing to the use of low-carbon dirty steel.

Again, several thousand pounds were saved by testing each weld by ultrasonic techniques. The circumferential and longitudinal welds were scanned and found to be free from defects. This particular vessel was 6 ft. in diameter and 10-ft. tall.

As a corollary to this work it can be expected that in the near future steel manufacturers will themselves examine thick plate by such techniques in a mechanised form. They will then be in a position to guarantee lamination-free plate within certain definite limits. It may be mentioned here that, while no definite standard testing specification exists either in Great Britain or America for ultrasonic testing, many important organisations are prepared to accept this test when it is done under the supervision of their inspectors.

Plant and Equipment

British firm to make line blind valves

During the past few years many plant engineers have felt the need to include line blind valves in their layouts but have had to revert to spectacle spades as line blind valves have not been available without dollar expenditure. The position has now been changed. Under an agreement with the Hamer Oil Tool Co., U.S.A., Joshua Hindle & Sons Ltd. will manufacture the full range of Hindle-Hamer line blind valves in the United Kingdom.

Line blind valves are said by the makers to provide a positive, reliable, easily operated shut-off which will reduce operating costs, product wastage, fire hazard and shut-down time. The reversing of a spectacle spade involves the slow and sometimes dangerous procedure of removal of bolts and nuts, spreading the line, knocking out the spade and replacement of packings, etc. This changeover occupies the personnel of the engineering or maintenance section for a considerable time and often means taking fitters from skilled work to do a job which the operating personnel could do themselves if line blinds were installed.

With a line blind valve in the line, the line can be blinded or opened in a few minutes by one person safely and positively. A handwheel or tommy-bar (according to the type of valve installed) is turned in one direction to free the reversible plate and a few turns in the other direction make a positive joint with the plate in its reversed position. A wide range of types is available ranging from the simple three-bolt type to the rigid-spool type.

The three-bolt type is a simply operated line blind in which the reversible plate is clamped between packings carried in the triangular body. This type is supplied in series 150, 300, 600 and 900 and is available with welding ends or flanged ends. Where it is desirable to avoid spillage and drainage of the line the 'fluid-saver' type should be installed. This also is operated simply by means of three bolts, but features an enclosed spectacle plate chamber which eliminates the necessity of draining the line and prevents spillage of the line fluid whilst the plate is being reversed.

For use on blending or distribution manifolds where lines must be opened and closed frequently and where space is often limited, the range of T and L line blinds are especially valuable.

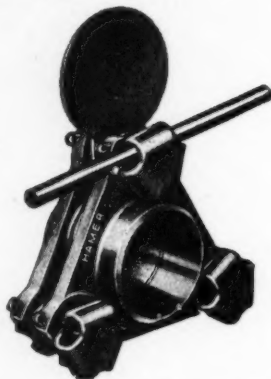
In positions where straight lines have to be opened or blinded frequently the handwheel-operated spool-type valves should be installed. This type is easy to operate and the spectacles can be reversed in a few seconds. An enclosed-plate chamber is incorporated, making it unnecessary to drain the line before the



The 'T' type line blind valve, for use on blending or distribution manifolds.

spectacle plate can be reversed. There is no spillage with its resultant product wastage and fire and accident risks, and pipe pits can be kept clean and tidy.

In all the types of line blind valves mentioned so far the operation of the handwheel or tommy-bar when releasing the spectacle plate involves a slight spreading of the line. In most installations this is readily accommodated by the expansion provided in the line to compensate for changes in pressure and temperature. There are, however, occasions when it is desired to blind a pipe at a point where no line movement is possible. Under these conditions, where it has in the past been impossible to use a spectacle spade, the difficulty can be overcome by the use of 'rigid' line blind valves. These employ a unique internal-expansion principle which eliminates spreading the line in even the slightest degree.



Line blind valve of 3-bolt type.

The operation is exactly the same as with the spool type; one man can open or blind a line in 1 min. The ball-bearing mounted ring gear controls the axial movement of a sliding internal sleeve which is keyed against rotation and therefore moves in a straight line to clamp or release the spectacle plate without requiring any spreading of the pipeline. The smoothly

operating ring gear is handwheel operated and a tremendous leverage is available for clamping the spectacle plate. Having a continuous one-piece body, the 'rigid' line blind valve is unaffected by misalignment, expansion or contraction of the line.

In certain circumstances it is necessary to install a single compact unit for blocking and blinding as an alternative to a line blind valve at the blinding point and a gate valve at the blocking point. For such a requirement line blind gate valves are available. These are a combination of the three-bolt type and either one or two gate valves. The single-valve-type line blind valve is used where line pressure is from one direction only, the gate valve end being placed on the upstream side. The double type allows the line to be blinded or opened under pressure from both directions, since closing both gate valves isolates the three-bolt line blind valve between them.

Many other types of line blind valves are available for special applications such as the four-bolt type, the geared type and the union type, etc.

Line blind valves are made to series 150, 300, 600 and 900 and are normally made in carbon steel, but when required they can be supplied in stainless steel or bronze. Three types of packing are available for all types.

Rod mills

As a result of an arrangement made with the Mine & Smelter Co., U.S.A., Marcy rod mills will be made by Pegson Ltd. of Coalville, Leicestershire. The mills will be available in three types:

- (1) Open-end mills, designed for wet or dry grinding of various ores and generally reducing $1\frac{1}{2}$ -in., 1 in. or finer feed to as fine as 35 or 48 mesh.
- (2) End peripheral discharge mills, specially advantageous where the production of fines is to be maintained at a low degree.
- (3) Centre peripheral discharge mills, designed to produce a minimum amount of fines. They are usually limited to a 4- or 8-mesh top size discharge product.

In the case of the open-end rod mills the area of the discharge opening is greater than that of feed entry, providing the necessary pulp gradient for rapid change of mill content. The fineness of grade and tonnage may be varied easily by altering the mill speed, rod charge or dilution. A remarkably uniform product with a small amount of fines results from the line contact between rods and also that between rods and shell liners. The rods actually exert a 'screening' action on the pulp, preventing the discharge of over-size.

The end peripheral mill is most widely used in the grinding of such materials as coke and friable non-metallics, materials for glass, pyroborates, and gravel for sand

production. The material to be ground enters through a standard trunnion and is discharged through port openings placed equally around the mill periphery. For wet grinding the mill emphasises the Marcy principles of grinding due to the steep gradient between the points of entry and discharge. The makers claim that under dry conditions no mill is so well adapted to produce a -4 or -8 mesh product in one pass with the production of a low amount of fines.

The centre peripheral discharge rod mills are constructed to allow for feed to enter at both ends and to be discharged at the centre by means of discharge ports spaced equally around the mill periphery. Primarily developed to produce sand to meet Government specifications, these mills have found wide use in grinding friable non-metallics, industrial minerals and ores which tend to slime easily. Wet or dry grinding is possible by changing the rate of feed, pulp dilution, rod charge and discharge port area. It is possible to produce sand of virtually any fineness modulus. Distinct advantages of this type of grind are that a cubical-shaped particle is produced and operating and maintenance costs are relatively inexpensive.

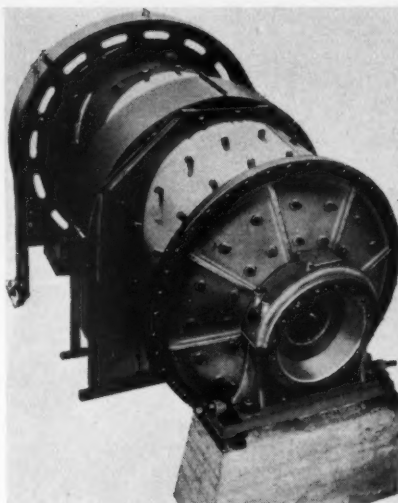
Hydrostatic contents control

A new hydrostatic contents controller and indicator has been introduced by K. D. G. Instruments Ltd. The general system and operation is similar to that of the hydrostatic contents gauge made by the same firm. This gives continuous reading at a distance without external source of power. The various transmitters fitted on the tank are also the same as in the hydrostatic contents gauge, with suitable units to meet general industrial, marine, chemical industry and other requirements.

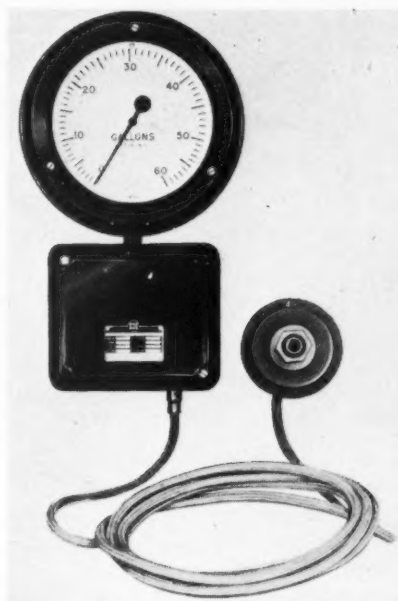
The controller is available with indicator-dial sizes of 7 in. and 10 in., the latter being suitable for either flush panel mounting or wall mounting, while the 7-in. version is available for wall mounting only. In the 10-in. size the relay equipment and switch are housed within the indicator case. Lights and cancel switches, etc., can be mounted on the lid of the case below the indicating dial. The 7-in. size is designed to have a switch unit attached below the indicator head.

In both cases the switch circuit can provide snap-action contact at one level only or, with the use of relay equipment, a two-level contact arrangement is available. A wide range of relay equipment can be offered similar to the range offered with the micro-pressure switch and liquid-level switch.

A feature of the controller is that the switching unit is independent of the indicating pressure element and the makers claim that true indication at all levels is guaranteed. Further, the switch contains precision setting with extremely small differential.



Rod mill with centre peripheral discharge.



Hydrostatic contents controller and indicator available with dial sizes of 7 in. and 10 in. The instrument gives continuous reading at a distance without an external source of power.

The use of two contacts together with relays results in an extremely flexible arrangement and permits a variety of tested circuits to be offered, e.g. lights indicating low, normal and high; alarms at high and low or at high and dangerously high

For further details of plant and equipment please use the coupon on page 302

levels. Alarms can be silenced by push button with automatic reset on return to normal or the low level can initiate switching action (e.g. a pump) to be reversed at the high level. Provision has also been made on the 7-in. version only for fitment to pressurised tanks with static pressures up to 30 p.s.i.

This type of instrument can, of course, be used for indication of pressure, vacuum, altitude, differential pressure, etc.

TV for remote water gauge indication

It is believed that the *Levelscope* is the first application of closed-circuit television for remote water-gauge indication commercially available in Britain, although a number of installations exist in the U.S.A.

With the increasing drum height of modern boilers, optical methods of water-gauge observation become inconvenient or actually impossible. Repeater gauges using electrical, hydraulic or mechanical transmission of various types have, of course, been available for many years, but suffer from the disadvantage that, no matter how clever their design or excellent their construction, the possibility of failure, although remote, cannot be entirely ruled out and the consequences of losing the drum water of a large boiler through a false indication are too serious to contemplate. Closed-circuit television *cannot* give a false reading; it shows either a true picture of the gauge or nothing at all.

The *Levelscope* consists of a simple camera unit with power pack incorporated, and a receiver unit mounted in some convenient position. In certain circumstances a 'rear cell' unit, clamped on the water gauge, may be necessary with some types of gauge. The transmitter and receiver are coupled with an ordinary co-axial cable, and the equipment uses components which are readily available commercially. No special lighting is required, the camera carrying its own built-in ultra-violet lamp, so that it can operate, if necessary, in total darkness.

The unit is being demonstrated at the Chemical Plant Exhibition, London, by Bailey Meters & Controls Ltd., on the stand of Babcock & Wilcox Ltd.

Flameproof barrier box

A flameproof barrier box for connecting conduit entering an inflammable atmosphere from a non-inflammable atmosphere, or *vice versa*, has been marketed by General Electric Co. Ltd.

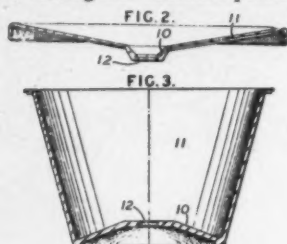
Fitted with four 20/25 amp. terminals it is suitable for use in circuits up to 660 v. and has two 1-in. e.t. outlets on both incoming and outgoing sides. It has been approved by the Ministry of Fuel and Power for use in gases of Groups II and III and has been awarded Buxton Certificate FLP 3027 (Nov. 1952). The price is £2 18s. 4d.

Chemical Engineering Invention

MONTHLY SUMMARY OF PATENT CLAIMS

Rotor for a centrifugal still

A rotor for a centrifugal still, and particularly for a high-vacuum unobstructed-path still wherein the distilland is in the form of a film, is specified. It is of massive construction, has a thickness at the vaporising area between $\frac{3}{16}$ and $1\frac{1}{2}$ in., and is made of material which has a coefficient of thermal conductivity in excess of 90 B.Th.U./ft./hr./°F. The temperature of the vaporising surface using such a rotor heated by an electric element beneath it is maintained nearly uniform at all points, and decomposition of a heat-sensitive distilland is less than when a thin rotor is used. For a rotor of 30 in. diameter, the thickness is in the range $\frac{1}{4}$ to $\frac{1}{2}$ in. The rotor may be made of aluminium, copper, gold, silver or magnesium, or various plated combinations of such metals. It may also be made of an alloy of sufficiently high thermal conductivity. A rotor constructed of copper with a silver coating has excellent performance

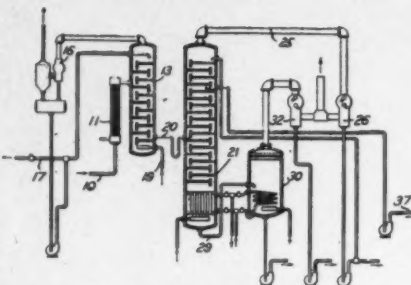


characteristics. Figs. 2 and 3 show rotors which are suitable for small and large stills, respectively. In either case the rotor is rotated by a shaft (not shown) attached at hole 12. The heating element underneath the rotor is placed in operation. The distilland is introduced at area 10, and centrifugal force drives it over the vaporising area 11 into collecting gutters. In an example, comparison is made between the performance of a $\frac{3}{16}$ -in. aluminium rotor and a $\frac{3}{16}$ -in. steel rotor in the concentration of vitamin A from fish oil. The loss of vitamin A using the thick aluminium rotor was about one-half the loss sustained when the thin steel rotor was used.—644,631, Distillation Products Inc.

Distilling tall oil

Tall oil is separated into a fatty acid fraction and a resin acid fraction by submitting the tall oil, preferably after removal of its low-boiling constituents, to fractionation under sub-atmospheric pressure. The main fatty acid fraction is withdrawn from the fractionation as a liquid side-stream, while the resin acid is withdrawn as liquid bottoms.

Tall oil was fed through line 10 into heat exchanger 11, and then introduced into fractionating tower 13 which was connected through condenser 16 with a



vacuum pump, and supplied with live steam through line 19. The top of the tower was maintained at 375°F. and 35 mm.; the bottom at 475°F. and 65 mm. The overhead vapours were condensed in condenser 16, and three-quarters of the condensate was returned to the tower as reflux. Tall oil, free from low-boiling constituents, was withdrawn from tower 13 through line 20, and introduced into fractionating tower 21. The top of tower 21 was maintained at 375°F. and 4 mm.; the bottom at 545°F. and 40 mm. The overhead vapours taken off through line 25 were condensed in condenser 26, and a large percentage of the condensate (equivalent to a reflux ratio of 14) was returned to the tower. The main fatty acid product was taken off the fourth tray from the top of the tower, and was withdrawn through line 37. Resin acid was withdrawn from the base of tower 21 through line 29 and introduced into flash chamber 30, which was maintained at 550°F. and 5 mm. The vapours from the flash chamber were condensed in condenser 32 to give the resin acid product. Both flash chamber 30 and tower 21 were supplied with small quantities of live steam.—640,244, Armour & Co.

Pelleting carbon black

In the pelleting of carbon black by agitating the black with a restricted amount of water, e.g. 20 parts per 20 parts of carbon black, with an agitator having surfaces which exert a sufficient compacting action to form pellets, the carbon black is subjected to a preliminary densing operation. This is achieved by agitation with an amount of water less than that required for pelleting, e.g. $\frac{1}{2}$ to $\frac{3}{4}$ of the restricted amount, with an agitator having surfaces which exert insufficient compacting action until the black is uniformly dampened.

Both treatments may be carried out in cylindrical vessel 4 having an S-shaped rigid member 2 with depending round rods 3 mounted on motor-driven rotating shaft 1, so that the furnace black is subjected to roller pressure. Preferably, however, rods 3

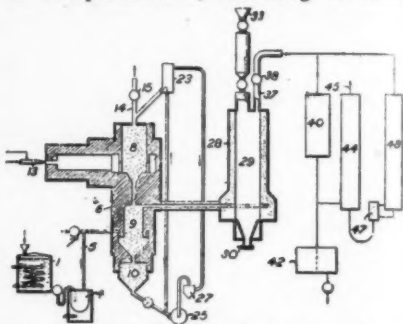


having thin knife or wedge-shaped forward edges are used in the preliminary operation. The water may be added all at once or in increments and the black may be de-aerated before treatment. Specification 640,565 and U.S.A. Re-issue Patent 21379 are referred to.—640,661, Columbia Carbon Co.

Carbon bisulphide manufacture

Carbon bisulphide is made by passing sulphur vapours, superheated to 600 to 800°C. by passage in countercurrent to and in direct contact with a moving bed of hot small particles of a refractory material, into contact with reactive carbon, e.g. charcoal, in an insulated reaction zone maintained at a temperature not materially exceeding 850°C. solely by the heat of reaction.

As shown, molten sulphur from kettle 1 is vaporised at 4 and the vapours passed to a superheater 6, consisting of three



refractory-lined chambers 8, 9, 10. Mullite pebbles, heated in chamber 8 by the combustion products of gas or oil burner 13, pass through chamber 9, wherein they heat the sulphur vapour, and thence to chamber 10 from which they are returned to chamber 8 by an air lift, the air separated at 23 returning to a blower 25 via a dust separator 27. The heated vapours pass into an insulated reactor 28 to react with charcoal from a hopper 33. Ash is removed periodically through outlet 30, while exit gases are passed to condenser 40, liquid carbon bisulphide passing to receiver 42. Non-condensed gases pass to oil scrubber 44, hydrogen sulphide escaping through line 45. The wash oil passes to reboiler 47 and stripper 48, whence the separated vaporised carbon bisulphide is returned to the condenser 40. To start the reaction, the charcoal in chamber 29 is burned by air admitted through inlet 7 until a glowing zone is established, when the air is shut off and a flow of sulphur vapour started. To prevent gas leakage from chamber 8 to chamber 9 or vice versa the static pressure in the two chambers is equalised by adjustment of valve 15 in vent stack 14 and valve 38 in exit pipe 37 of the reactor.—642,557, Dow Chemical Co.

World News

GREAT BRITAIN

New company to build petroleum catalyst plant

Johnson Matthey & Co. Ltd. have formed a subsidiary for the manufacture of catalysts in conjunction with Universal Oil Products Co., Illinois, U.S.A. The new firm, Universal-Matthey Products Ltd., was incorporated on July 9. It will manufacture and make available in the sterling market U.O.P. Platforming catalyst for use by refineries in producing premium-quality petrol. Among the first refineries in the U.K. to be supplied with the new company's catalyst will be those of the Anglo-Iranian Oil Co. Ltd. and Shell Refining & Marketing Co. Ltd.

Universal-Matthey has offices at 78 Hatton Garden, London. Mr. L. C. Montague, a managing director of Johnson Matthey, is chairman and Mr. C. D. Sims of Universal Oil Products is general manager.

A large plant to manufacture the Platforming catalyst will be built in England; it is expected to go into production in mid-1954.

Platforming—developed, engineered and licensed by Universal Oil Products Co.—is a radically new petroleum-reforming process that changes the chemical structure of petrol having a low octane number to yield high octane material. This is done by passing the inferior grades of petrol together with hydrogen at high temperature and high pressure over a catalyst containing platinum. The Platforming catalyst acts only on the part of the petrol that needs octane improvement. In the process corrosive sulphur compounds and gum-forming compounds are removed.

In addition to upgrading inferior petrols, Platformers also produce aviation spirit base stock and aromatic hydrocarbons—benzene, toluene and xylenes—which are finding new uses in the petrochemical industry.

Coal processing research

A 50-gal./day pilot plant for the synthesis of liquid fuels and chemical products from coal by the Fischer-Tropsch process is in operation at Greenwich, London. In this process the desired products are obtained by the chemical reaction of carbon monoxide and hydrogen (prepared by the gasification of coal) in the presence of a solid catalyst.

A small experimental plant employing a liquid-phase technique for carrying out and controlling the reaction is the main exhibit on the stand of the Department of Scientific and Industrial Research at the Engineering, Marine and Welding Exhibition, London. The gas mixture is passed through a suspension of powdered iron catalyst in molten wax maintained at 265°C. and a pressure of 20 atm. The

products are recovered by cooling the exit gas stream. Models illustrating other methods of working this process, with fixed and fluidised beds of catalyst, are also being shown.

Included in the exhibits illustrating some aspects of the work of the British Hydromechanics Research Association is a transparent working model showing how coal can be transported hydraulically. In this plant coal passes through a mechanical feeder situated in the mine, working into a vertical pipe, and is raised to the surface by upward-flowing water. Model components are approximately one-eighth full size and are circulating nearly 10 cwt. of coal and 400 gal. of water per hour.

The chemical industry and the B.I.F.

The following statement has been issued by the Association of British Chemical Manufacturers:

'Critical comment has been made on the declining interest shown both by exhibitors and buyers in the Chemical Section of recent British Industries Fairs, and of the rapid decrease in the size of the section. . . . It is clear that the present arrangements are unsatisfactory and that a new approach is needed. To give time for proper consideration of a long-term policy, therefore, the Association has decided not to organise a Chemical Section at the 1954 British Industries Fair.

'To avoid misunderstanding, the Association wishes to make it clear that this temporary withdrawal does not mean that the chemical industry has decided to leave the Fair permanently. The sole object of this blank year is to provide a breathing space, during which time the Publicity Committee and the Chemical Section Advisory Committee will examine ways and means of organising a satisfactory and comprehensive display in future years. Such displays may well be at the British Industries Fair in London or Birmingham, and either alone or in co-operation with allied industries such as plastics and chemical plant. . . .

'There is almost unanimous agreement, however, on the need to stay out of the 1954 Fair to allow proper consideration of long-term policy. Another partial showing like 1953, labelled as a Chemical Section, might well destroy any possibility of a successful section in future Fairs. . . .

'This statement is issued to make the Association's position and intentions clear, and in the hope that potential chemical exhibitors will act in concert with members of the Association in the matter.'

Change of name

Structural & Mechanical Development Engineers Ltd., the construction company of the Almin group, have changed their name to S.M.D. Engineers Ltd.

Underground gasification experiments

The results of experiments on the underground gasification of coal in Britain are encouraging, according to the Minister of Fuel. More than 1,500 tons of coal have been gasified and much useful information obtained.

Experiments have been in hand since 1950, and work is now proceeding at two sites: Newman Spinney, near Sheffield, and Bayton, Worcestershire. The experiments are designed to establish the practicability of starting large-scale operations and to provide the data necessary for the planning of such operations.

Underground gasification is discussed in an article elsewhere in this issue.

Rubber lining for 24-million-gal. reservoir

What will certainly be the biggest rubber-lining job ever attempted has been started at Easington, Durham. There the Mill Hill reservoir, with a capacity of 24 million gal. and measuring 750 ft. long, 300 ft. wide and 18 ft. deep, is leaking badly because of subsidence caused by mining operations. Consequently, its capacity is only 3 million gal. Caulking with pitch, cement and plastic has been only partially successful, so the Sunderland and South Shields Water Co. has made the bold decision to invite the Dunlop Rubber Co. to give the whole reservoir a rubber lining. Dunlop first proved they could do the job by lining the most difficult corner, for which they evolved a method of bolting sheets of $\frac{1}{4}$ in. covering to the walls and fitting a glove round the pipe. The cutting-out of the rubber for the complete job is being done in the firm's Manchester factory.

Cheaper aluminium-coated steel

Development of a 'hot dip' method of coating steel with aluminium is reported in the second annual survey of the British Iron and Steel Research Association.

Aluminium-coated steel sheet is regularly produced in the United States, but few British plants can install the equipment without heavy capital expenditure. The new process, however, reduces the capital cost.

Another new coating method, the report says, is being developed by the simultaneous depositing of iron and zinc on steel sheet, the zinc content improving corrosion resistance. Similar coatings that have the brightness of a mirror can also be used for ordinary galvanised goods such as roofing sheets, wire and hollow ware.

Telephone number change

The telephone number of the Association of British Chemical Manufacturers has been changed to Hyde Park 4126/7 and 1557/8. This change also affects seven affiliated associations operated from the same address.

Standardisation of process control instruments

The British Standards Institution has issued recommendations for certain of the dimensional features of industrial measuring and control instruments and of the panels in which they are mounted.

These recommendations are intended to encourage the adoption of this principle of interchangeability and to ensure satisfactory operation in cases where items of equipment complying with the recommendations are connected together, even when they have been made by different manufacturers.

The recommendations specify the dimensions of circular and strip-type charts for recorders, and give the overall dimensions and mounting dimensions for clocks for circular-chart and drum-type recorders, together with the appropriate drum sizes for the latter; they indicate the appropriate sizes of screw threads for the measurement connections and pneumatic connections to instruments and give guidance on the air-pressure range for automatic control and remote indication or recording instruments.

Recommendations are given regarding the sizes and constructional features of panels and sub-panels and for the aperture sizes for instruments with circular and rectangular cases, and guidance is given as to the manner of fixing instruments to panels.

Copies of this standard (B.S. 1986: 1953) may be obtained from the B.S.I., 2 Park Street, London, W.1, price 3s.

British Standard Institution's new headquarters

The British Standards Institution is now in full operation at its new premises at 2 Park Street, W.1. The new building will contribute to more efficient working by concentrating the B.S.I.'s scattered departments under one roof. It will also provide increased and more convenient accommodation for the 13,000 specialists who attend the 3,500 B.S.I. committee meetings held during the course of each year.

Crosfield's new chairman

Dr. J. E. Taylor, B.Sc., Ph.D., has been appointed chairman of Joseph Crosfield & Sons Ltd. in succession to the late Mr. P. A. W. Came.

Dr. Taylor joined British Oil & Cake Mills Ltd. at Selby in 1934 and subsequently was engaged with the Technical Division of Unilever Ltd. He joined the board of Joseph Crosfield in 1952. He has also been appointed chairman of William Gossage & Sons Ltd., another post held by Mr. Came.

Mr. Came died suddenly on June 24. Besides being chairman of Crosfield's and of Gossage's and a director of Industrial Soaps Ltd., he was also chairman of the Oil Hardeners' Committee, a member of the General Council and chairman of Group E of the Association of British Chemical Manufacturers and a member of the Joint Industrial Council of the Soap,

Candle and Edible Fat Trades Employers' Federation. He was a member of the North Western Regional Board for Industry from May 1946 to May 1947.

Styrene Copolymers staff changes

Mr. R. S. Robinson, Chief Chemist at Styrene Copolymers Ltd. for the past five years, has left the company to take up residence in Canada for reasons of health.

Mr. W. R. Moon, Sales and Technical Service Manager since soon after the company was formed, and who has worked very closely with Mr. Robinson, has now taken over from him the position of Chief Chemist. Mr. Moon is a Manchester man and he graduated at the Manchester College of Technology. He was with British Titan Products Co. Ltd. before coming to Styrene Copolymers.

Mr. D. J. I. Davies has joined the company as Sales Manager. Mr. Davies is a Jesus College, Oxford, graduate and he served during the war in the Royal Engineers. After experience as a chemical engineer with I.C.I. (Billingham) and the Mond Nickel Co. Ltd., Mr. Davies joined L. A. Mitchell & Co. Ltd., Manchester. There he was concerned with the design of alkyd resin plants and their sale to paint manufacturers.

Costain-John Brown appointments

Mr. C. C. Bates has been appointed chief welding engineer of Costain-John Brown Ltd. In addition he will be responsible for the technical supervision of Welding Supervision Ltd., their associate company. Mr. Bates, who is 39, joined Costain-John Brown Ltd. from the British Welding Research Association, where for the past four years he was one of the senior development engineers responsible for helping industry to apply the results of research, and member firms with design, technique, metallurgical and inspection welding problems.

Sulphur and acid price control ended

The Minister of Materials has made orders freeing sulphuric acid and ground sulphur from price control. The orders which provide maximum prices for sulphuric acid and ground sulphur were revoked on August 4, 1953.

Acid statistics

Production of sulphuric acid in the U.K. for the second quarter of 1953 totalled 459,859 tons, 81.3% of capacity. Consumption totalled 461,295 tons. The stock at June 30 was 63,076 tons, compared with 59,020 at April 1. Stocks at June 30 of all raw materials excepting anhydrite were below stocks at April 1.

Mirrlees Watson's new orders

Two substantial contracts for Canada—a stainless-steel triple-effect evaporator for corn steep liquor and a grain distilling plant for potable spirit—were booked by Mirrlees Watson Co. Ltd. recently. Other new orders listed in the chairman's review of operations for the year include a 15-roller

36 × 78 in. turbine-driven sugar mill for St. Kitts in the British West Indies and a complete new factory in Venezuela to mill 2,500 tons/day of cane and produce refined white sugar.

Three 42 × 84 in. mills and a Searby shredder supplied to Natal (South Africa) are now operating, and a further order has been received for one more mill of similar size to make a complete train of 18 rollers.

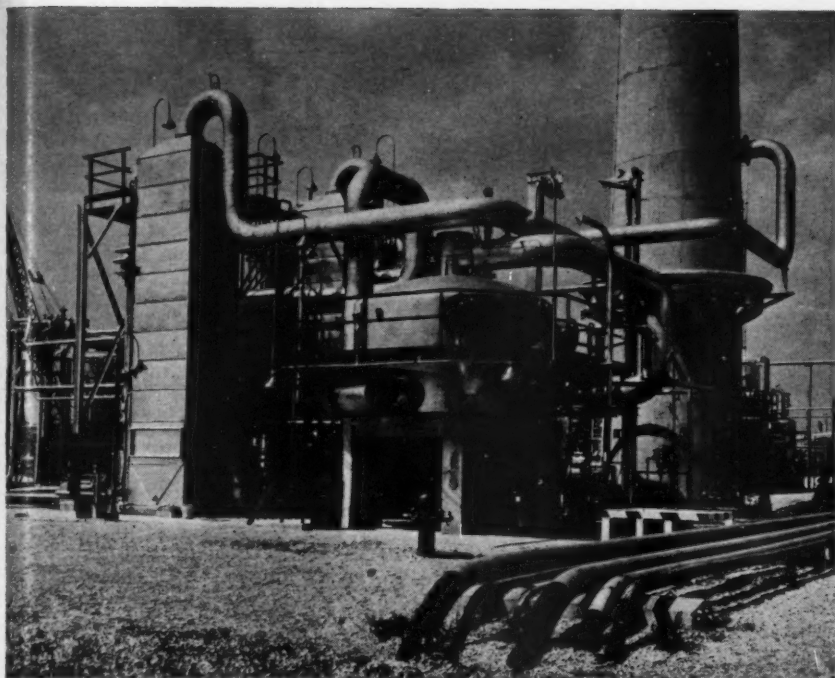
The supply of sugar machinery to the Central Rio Haina factory in the Dominican Republic is nearing completion in spite of delays. The new machinery is being erected as an extension to double the capacity of the existing factory and should be in service during the coming milling season.

Mr. W. Leonard Hill entertains S.C.I. members

Members of the Food and Agriculture Groups of the Society of Chemical Industry were guests of Mr. W. Leonard Hill and Mrs. Hill at a garden party held on August 1 at their home, Crux Easton, near Newbury. Mr. Hill, who is chairman of Leonard Hill Ltd., publishers of CHEMICAL & PROCESS ENGINEERING and 11 other technical journals, is this year's chairman of the Agriculture Group. About 80 guests attended the garden party, which was favoured with brilliant sunshine, and an extremely pleasant afternoon was spent in the beautiful grounds of Crux Easton where guests were able to see some of the work in progress on Mr. Hill's farm, including the reclamation for agriculture of a considerable area formerly used for cottage gardens.

New viscosity standard

On July 1 the British Standards Institution adopted the new value of 0.01002 poise for the absolute viscosity of water at 20°C. An amendment (P.D. 1655) to the fundamental British Standard on viscosity determination (B.S. 188: 1937) has accordingly been issued. The new value has recently been obtained by the National Bureau of Standards, U.S.A., using improved methods of viscosity determination, and it replaces the hitherto adopted one of 0.01005 poise. It has been agreed between the B.S.I. and the American Standards Association that the new value should be accepted for standards as from July 1. This may have an impact on the work of several industries. For example, in the petroleum industry it is customary to use viscometers which have been calibrated with water and such viscometers bear a factor by which the experimental observations are multiplied in order to determine the viscosity of the oil under investigation in absolute units. This factor is based on the value for the viscosity of water which, at the time of calibrating the viscometer, has been accepted as being the most accurate. Obviously the introduction of a new value for this viscosity of water will affect the factors on instruments used by the petroleum industry.



NEW SULPHUR RECOVERY PLANT

This plant, recently put to work at the Fawley petroleum refinery of Esso Petroleum Co. Ltd., produces 40 tons/day of 99.5% pure sulphur from gases generated in the fluid catalytic cracking process. The plant, referred to in an editorial note in this issue, is one of four refinery projects for increasing the production of sulphur in Great Britain.

AUSTRALIA

Sulphuric acid plant will raise fertiliser output

Demands of agriculture in all states are increasing the need for sulphuric acid supplies for fertiliser manufacture. To meet the deficiency, a new sulphuric acid plant designed to use local pyrites is to be built at Cockle Creek, south of Newcastle, N.S.W., at a cost of £A1 million. The plant capacity will be 100 tons/day. Local steel will be used for all structural purposes and machinery will be imported from the U.K.

Coal preparation plant ordered

General Electric Co. Ltd. has received a contract to provide a coal preparation plant for the Corrimall colliery, near Port Kembla, 40 miles south of Sydney, N.S.W.

This plant is to have a capacity of 200 tons/hr. of coking coal, and the equipment includes one 10-ft. diameter Chance cone with ancillary screening and loading equipment which will comprise Sherwen electromagnetic screens and Fraser & Chalmers shaker-type screens.

A water-clarification plant is also to be supplied.

Cellulose acetate factory

The new factory of C.S.R. Chemicals Pty. Ltd., at Rhodes, was opened by the Governor-General, Sir William Slim, last month. The factory, built at a cost of £5½ million, will produce chemicals not previously made in Australia. C.S.R. Chemi-

cals is a joint enterprise of the Colonial Sugar Refining Co. and the Distillers Co., of England, and is pioneering the manufacture in Australia of a group of organic chemicals.

The main product at the new plant is cellulose acetate for the production of acetate rayon by Courtaulds (Australia) at its new factory near Newcastle.

Some of the cellulose acetate will be further treated at Rhodes to produce basic material for making a large range of plastic articles.

Other organic chemicals being produced include raw material for aspirin, vitamin C and acetic acid.

Atomic energy hopes

The Premier of South Australia, Mr. Playford, said recently that the state's next power station, which would be needed by about 1960, would be operated by atomic energy. He added that it would follow the completion of two coal-burning stations now being constructed at Port Augusta.

Mr. Playford, who has recently inspected atomic energy research stations in Britain and the United States, said he was convinced that his forecast made eighteen months ago that the state would have industrial atomic power within ten years would prove conservative. He emphasised that the atomic power which would be available in 1960—from more advanced and efficient atomic piles—would be cheaper in South Australia than power from present fuels. Britain, he declared,

was now seriously challenging America's superiority in the sphere of atomic energy for commercial and domestic purposes.

South Australian generating stations now use either coal from New South Wales or low-grade fuel which must be brought long distances by rail from the state's own coal deposits. At Radium Hill, the state has Australia's most advanced plant for the recovery of uranium.

NEW ZEALAND

Heavy-water project may use geothermal steam

Dr. H. R. C. Pratt, who is in charge of the heavy-water project at the atomic energy research establishment at Harwell, has visited New Zealand with two other British consultants to make the final report on which the two Governments will decide whether to proceed with the production of heavy water from geothermal steam at Wairakei. In an interview, Dr. Pratt said his party had been gathering information on labour and materials required for an accurate forecast of costs.

Although he declined to elaborate on proposals for Wairakei, Dr. Pratt spoke generally on the production of heavy water. The concentration of heavy water in ordinary water had been correctly described as 'one drop in a cupful.' The actual figure was about one part in 7,000. An enormous amount of steam was required and the geothermal output of Wairakei was a promising source.

A new type of distillation column developed at Harwell had improved the economics of heavy-water production. 'It is more compact, but it is still pretty big,' Dr. Pratt said. The plant for producing heavy water, with its tall columns, would resemble an oil refinery.

Asked whether the export of heavy water presented any problems, Dr. Pratt said this was the least of the difficulties. Heavy water was harmless and could be transported in ordinary drums provided they were strong enough to withstand accidents which might cause wastage of a product valued at something over £40/lb.

If the heavy-water project at Wairakei was approved, there was no reason why its design, erection and operation should not be undertaken by New Zealanders, Dr. Pratt said in answer to another question. Such a project would undoubtedly open up new opportunities for chemical engineers in New Zealand.

Dr. Pratt came to Christchurch to visit the chemical engineering department of Canterbury University College. This is the only department in the University of New Zealand exclusively devoted to the training of chemical engineers. Special studies were first provided in 1944 as part of the chemistry department. In 1950 chemical engineering became a separate department of the National School of Engineering.

As the only training centre in New Zealand, the department had an important

place in projected developments, Dr. Pratt said. He spent most of today with the lecturer-in-charge, Mr. S. R. Siemon, the senior lecturer, Dr. T. Hagyard, and students of chemical engineering. He inspected the new 35-ft. 'spinning cross' distillation column in which the department has introduced a new mixing method for the production of heavy water. Later he addressed students.

Scheelite production

The largest and most valuable shipment of scheelite to leave New Zealand since the end of World War 2 was recently shipped to America. The consignment weighed 27 tons and was valued at £25,000.

Though there are other deposits of scheelite in the Dominion, the only mines being worked are the one at Glenorchy, at the head of Lake Wakatipu, and that at McRae's Flat, near Palmerston, in the far south of the South Island of New Zealand.

The scheelite produced is of a high quality and compares more than favourably with that from China, Portugal and Burma. Consequently it has been eagerly sought in both the United Kingdom and the United States.

The price of the product can in a way be regarded as a barometer of world affairs. Immediately after World War 2 there was a drastic slump in the price, but, with the outbreak of the Korean war, there came a spectacular increase and the price soared to about £2,000/ton. The situation appears to have eased a little and with more supplies becoming available the value has dropped to about £1,000/ton and it is expected to decrease still further.

The British Government, realising the urgency for supplies, set up a buying agency to gather all possible supplies for Britain and most of New Zealand's output has been sent there. The average production in New Zealand is about 40 tons, but during World War 2, when the Glenorchy fields were operated by the Mines Department, the highest production for one year was 130 tons.

Scheelite is a source of tungsten, though not so important as wolframite.

SOUTH AFRICA

Cement from blast-furnace slag

The South African steel-making concern, ISCOR, has set up a pilot plant for producing cement from blast-furnace slag, the Council for Scientific and Industrial Research has announced. ISCOR and Vanderbijl Park produce about 2,000 tons of slag a day, from which some 20,000 pockets of cement could be made (one pocket equals 94 lb.).

Essential oils factory

Oceanic Refineries (Pty.) Ltd., Wychwood, Johannesburg, is a new company formed to process and distil essential oils. A new factory has been built for this purpose. Initially the two main products will be roses and jasmine, but the company

is considering plans to produce avocado pear oil.

The new factory has been equipped with ultra-high vacuum distillation plant, which has been fabricated to the company's specification by the Versatile Engineering Corporation, also of Wychwood. Special stainless steel was used.

New salt plant

The Vraný Chemical Corporation Ltd. is setting up a £300,000 salt factory near Port Elizabeth. The plant, which will have a capacity of 100 tons of table and industrial salt per day, is scheduled to go into production by the end of this year.

This new venture is expected to save South Africa a considerable sum in foreign currency formerly used for imports of salt.

CHILE

Synthetic nitrogen plants threaten nitrate exports

The outlook for the Chilean nitrate industry's exports to its South American neighbours is threatened by reports that a number of them are planning the erection of synthetic nitrogen plants.

With Brazil, Chile had a convention providing that the former would refrain from building a synthetic plant and the latter would maintain in Brazilian territory a nitrate stockpile adequate to cover the country's needs of about 60,000 tons a year and any emergency. Last year, however, on renewing the convention, Brazil added a clause stipulating that no such plant would be constructed as long as no other South American country did so. Colombia recently announced her intention of building a nitrate plant and the Brazilian authorities immediately ordered the existing blueprints for a plant in São Paulo to be brought up to date.

Argentina is also reported to be planning a synthetic nitrate plant in Rio Cuarto to cover her requirements of 15,000 tons annually.

CUBA

Nickel plant to be enlarged

The U.S. Government has announced that it intends to increase the production of the Nicaro nickel plant in due course by 75%. The rate is now over 2 million lb./month, though the present target of 2.5 million has not yet been reached. The Bank of Agricultural and Industrial Development has obtained under Point Four an expert from the United States Bureau of Mines to advise on mineral development plans over the next two years.

Oil search

Two Cuban companies, one with half its capital derived from U.S. sources, are at present prospecting for oil, and the National Development Commission will provide the most promising with a drilling rig worth \$500,000 which it purchased last year. The Cuban petroleum law is to be revised in order to encourage further exploration.

INDIA

Indian Development Council for heavy chemicals

With the responsibility of advising the Government on the development of the country's heavy chemical industries, which include the manufacture of acids and fertilisers, a special Development Council has been set up in India.

Constituted under the Industries Development and Regulation Act, 1951, the new council held its inaugural meeting at New Delhi on July 10, at which Mr. T. T. Krishnamachari, Minister for Commerce and Industry, presided.

Saying that this was the second Indian Development Council to be set up, the Minister outlined the duties of the council, which cover, among other things, the expansion of particular units, general efficiency, utilisation of existing capacity, modernisation of plant wherever necessary and further development of the industry.

The council decided to collect basic information about the heavy chemical industries for their initial investigations. They discussed the present position of the acids and fertiliser industries in India and considered undertaking a survey of the rated capacity for the manufacture of some of the important chemicals and fertilisers.

Quinine factory for Assam

A quinine factory is to be built at Shillong (capital of Assam) under the five-year plan at an estimated expenditure of 500,000 rupees. The plant is expected to produce 10,000 lb. of quinine p.a.

NORWAY

Pelleting unit planned

A pelleting plant, the first in Norway, is to be built this winter at the Sydvaranger iron ore mines near Kirkenes. By 1957 it is hoped that about half of the mines' output of 1 million tons p.a. of ore can be turned into pellets. These command a better price than ordinary iron ore. In the first half of this year the Sydvaranger mines produced 400,000 tons of ore, of which slightly more than 300,000 tons were exported. The mines employ 985 persons.

TURKEY

Fertiliser project cancelled

The Etibank of Turkey recently called for tenders for a sulphuric acid plant at Murgul and a superphosphate plant at Hopa. This was reported in *CHEMICAL & PROCESS ENGINEERING*, April 1953.

The British Embassy at Ankara has now been informed by Etibank that it has been decided not to build the superphosphate plant. This is because another concern, which is shortly to build a superphosphate plant at Iskenderun, Turkey, is interested in buying the sulphuric acid to be produced at Murgul, and an agreement to this effect has already been made.

CANADA

Canadian acceptance of British pressure vessels

In view of the difficulties which have been experienced by British manufacturers in exporting pressure vessels to Canada, towards the end of last year the British Standards Institution had some discussions on the subject with Mr. La Bissonniere, the Chief Inspector of the Boiler Inspection Department of the Province of Quebec. As a result of discussions, Mr. La Bissonniere indicated that:

(1) He would accept a certificate from Lloyd's indicating that the welders engaged on the welding of a pressure vessel exported to Canada had been examined and had satisfied the welding tests approved by the Chief Inspector.

(2) He would accept a signed certificate from any of the recognised engineering insurance companies who are in the Associated Offices Technical Committee, and/or any competent inspection agencies that may be approved by the Chief Inspector, stating that any vessel which was exported to Canada complied with the requirements of the C.S.A. Code B-51 and the Province of Quebec Act and Regulations.

This arrangement with Mr. La Bissonniere related only to the Province of Quebec. The B.S.I. accordingly wrote to the Canadian Standards Association to ask whether it would be possible for similar arrangements to be accepted by the other provinces. The B.S.I. have now been informed that, at a meeting convened by the C.S.A. Committee B. 51 on Canadian Boiler Regulations, it was unanimously agreed that the arrangements made with the Province of Quebec would be acceptable in all the provinces of Canada.

Further information is available from Mr. J. P. Roberts, B.S.I., 2 Park Street, London, W.1.

BRAZIL

Newsprint production plans

The Brazilian Government is studying the possibility of producing newsprint and chemical pulp from local timber, according to official sources. Plans have been examined for the establishment of three paper and cellulose plants, which will be located at Marajo Island, at the confluence of the Madeira and Amazon Rivers and at a site above Manaus.

The Agronomy Research Service of the Ministry of Agriculture has sent 8 tons of white wood from the Amazon region to France for use in experiments in a French mill using a method known as the 'Iso-grand' method. Other samples were sent to the United States by the Northern Agronomy Institute for technological examination in that country.

Brazil at present spends around 30 million U.S. dollars a year on imports of newsprint and cellulose.

The Leonard Hill Technical Group—September

Manufacturing Chemist—Particle Sizes for Additives to Animal Foods; Analytical Determination of Thioglycolic Acid; Vitamins, 2; Chemical Plant Exhibition; Progress Reports on Detergents and Antibiotics; Ionones.

Food Manufacture—A New Yeast Factory; The Theory and Practice of Gas Packaging of Foodstuffs.

World Crops—Forestry in Canada; Robusta Coffee; The Harvesting of Fibre Flax Crops in Australia; Cane Sugar in East Africa.

Fibres—Detergents in the Wool Textile Industry; The Supply of Chemicals for the Textile Industry; The Chemistry of Wool Keratin; Chemicals for Use in Synthetic Fibre Manufacture; Oiling of Woollen Blends; Fibres for Pulp and Paper Manufacture, 2.

Atomics—Geiger and Proportional Counters in X-ray Diffraction Studies.

Petroleum—Fire Research, 1952; Fire Precautions in Oil Installations; Developments in Vaporising Liquid Fire Extinguishers; Modern Fire Safety Measures for Refineries and Tank Farms.

Building Digest—Rebuilding Western Germany, 2; Settlements in Buenos Aires.

Muck Shifter—Air Entrained Concrete; Harbour Work at Banias.

Paint Manufacture—Colours and Varnishes at the 31st Milan Fair; Congress of Industrial Chemistry, Paris; A New Non-destructive Test for Welded Varnish Plant; How to Solve Materials Handling Problems in the Medium-sized Paint Factory.

MEXICO

British machinery for bagasse pulp plant

More than 9,000 metric tons of pulp, made from bagasse (sugar-cane waste), will be produced annually in Mexico, according to the National Association of Exporters and Importers. Machinery and equipment has been bought from the U.K. It is being installed at Ayotla, in the state of Morelos, near the La Abeja sugar mill which will supply about 50 tons of bagasse daily.

Mexican imports of pulp amounted to 43,351 metric tons in 1951 and 44,246 tons last year, according to the Secretariat of National Economy. Although the demand for pulp is expected to continue to increase year by year, it is anticipated that the Ayotla will supply about one-fifth of the demand beginning early in 1954.

COLOMBIA

Fertiliser project

A Colombian company, Empresa Colombiana de Fertilizantes, was recently formed with the object of producing nitrogenous fertilisers from natural gas. The company has an authorised capital of 20 million pesos, of which to date 10 million pesos have been subscribed and 3 million pesos paid.

The company is now determining what

equipment will be required in order to start production and a survey of these technical requirements is being carried out by the Colombian Institute of Industrial Development.

U.K. firms who wish to offer their services as consultants or to sell their equipment should approach the Institute direct. The address is Instituto de Fomento Industrial, Avenida Jimenez No. 8-74, Bogota, Colombia.

JAPAN

Babcock & Wilcox venture

A new joint company has been formed to take the place of Toyo Babcock K.K. The new company, Babcock Hitachi Kabushiki Kaisha, has been established in Tokyo.

European know-how for silicones project

A technical contract is planned between the Japanese firm Shinetsu Chemical Co., the German Bayer company and the French chemical concern Rhone-Poulenc, it is learned in Paris.

The agreement is to provide for the exchange of patents between the companies to enable the Japanese firm to produce silicones. The Japanese company is to build a plant capable of producing 8 tons of silicones monthly, or enough to meet Japan's internal needs.

UNITED STATES

Hydrazine provides new field of chemistry

The Mathieson Chemical Co. has opened a \$3-million plant at Lake Charles, Louisiana, for the large-scale commercial production of hydrazine. Hydrazine, made from ammonia, is a rocket-fuel chemical which is said to provide a higher amount of energy per unit of weight than any substance known. Hydrazine is expected to form the base for an entire new field of chemistry, including the making of dyes and synthetic detergents. Mathieson will make a 95% pure product. When first sold in the U.S.A. the price was \$50/lb., but Mathieson's pilot plant brought the price down to \$9 in 1948, and the current price is \$2½. With further process improvements, the company hopes ultimately to cut the price to 50 cents/lb.

Titanium dioxide project

American Cyanamid Co.'s Calco Chemical Division plans a \$14-million titanium dioxide plant on the outskirts of Savannah, Georgia. The plant will be the division's third for production of this material. Construction will start in the last quarter of 1953 and completion is scheduled for early 1955.

More 'Freon' to be made

E. I. du Pont de Nemours & Co. Inc. has announced a \$2-million expansion programme at two plants manufacturing Freon fluorinated hydrocarbon compounds

for the refrigeration and aerosol industries. Major part of the new manufacturing facilities will be located at the Chamber works plant of the Organic Chemicals Department at Deepwater Point, New Jersey.

The expansion programme reflects the rapid growth of both the refrigeration and aerosol industries, which use *Freon* compounds as cooling agents or propellents.

World's largest 'platformer' for Texas refinery

The world's largest 'platforming' unit is now in operation at Shell's Houston, Texas, refinery. This new plant has been designed to produce either petrol or benzene and toluene. Production is at present confined to benzene and toluene—16 million gal. p.a. of benzene or 28 million gal. p.a. of toluene—and no petrol manufacture is contemplated in the immediate future.

There are three phases to the refinery's processing, of which the initial one is the feed preparation. Light naphthas are distilled from east and west Texas crudes, then blended with a selected naphtha cut. The feed-stock mixture is charged to the 'platforming' feed preparation unit and other similar units, and fractionated to provide narrow-range feed stock containing nearly all of the C6 and C7 fractions.

Feed stock is then charged from the fractionators to the 'platformer,' where the catalyst which contains platinum reforms components of the feed stock into aromatics.

Reactor effluent is separated into a liquid and a gas stream. Part of the gas stream is vented to refinery fuel through an absorber to recover light hydrocarbons, including benzene. The rest is recycled to the process through a gas scrubber to remove hydrogen sulphide.

The third step in the refinery's process consists of depentanising the liquid stream and charging it to the aromatic-recovery unit, where the aromatic hydrocarbons are separated and purified.

Giant uranium plant nearly complete

The U.S. Atomic Energy Commission's \$78-million plant at Fernald, Ohio, for converting uranium ore into fissionable feed for other A.E.C. plants, is coming into operation as it nears completion. Three hundred sub-contractors participated in the construction work, the total cost of which will be about \$47 million. At the peak of construction in March 1952, 3,200 workers were employed.

According to *Chemical Engineering*, July, by completion about \$30.4 million will have been spent on the engineering part of the project and in procuring process equipment. Much of the equipment had to be specially designed and more than 1,200 firms were involved. A precision mill had to be designed to roll uranium ingots into bars in production quantities. Special furnaces, tanks, vessels and conveyors were required. In addition, power centres, boilers, electric motors and control centres had to be procured.

The size of the present project is the outcome of considerable additions to the original specifications, which were for a \$30-million undertaking. Acquisition of the 1,050-acre site alone cost \$600,000.

The Feed Materials Production Centre, as it is called, is a completely integrated series of plants whose function is to convert raw uranium ore into highly-pure uranium metal in various shapes. The process is designed to reclaim essentially all of the uranium contained in the ores and also much of the chemicals used in the processing steps. Among intermediate products in the process are orange oxide (UO_3), green salt (UF_4) and metal ingots, rods and slugs.

Manufacture of uranium feed compounds and metal, as at Fernald, is an integral part of the chain of processes culminating in production of fissionable materials. The fissionable materials and other isotopes produced from the products of the Fernald or similar plants, can be used for military or peace-time purposes.

Acrylonitrile expansion

Growing demand for acrylonitrile for use in the manufacture of synthetic rubber, plastics, fibres, soil conditioners, etc., is bringing into being a number of plans for expanded output. By 1955, output capacity is expected to reach 200 million lb., against 50 million in 1952 and about 17 million in 1950. Price reductions are expected to accompany higher production, with some sources predicting a cut of as much as 30% on present levels by 1955.

The B.F. Goodrich Chemical Co. plans to build an \$8½-million plant at Calvert City, Kentucky, to manufacture acrylonitrile. The new plant is scheduled to be in operation by the autumn of 1954. It will be the company's second factory at Calvert City—the first one being a recently opened \$6-million chloride monomer plant.

MEETINGS

Incorporated Plant Engineers

September 10. 'Maintenance Costs,' by H. Whitfield, 7 p.m., Roadway House, Oxford Street, Newcastle-on-Tyne.

September 16. 'Education and the Plant Engineer,' by H. N. Henry, 7 p.m., Scottish Building Centre, 425/427 Sauchiehall Street, Glasgow, C.2.

September 21. 'Paint in Industry,' by P. I. Gay, 7.15 p.m., Radiant House, Bold Street, Liverpool.

September 24. 'Electrical Factory Regulations,' by H.M. Factory Inspector, 7.30 p.m., Grand Hotel, Sheffield.

International Conferences

September 7-11. Conference of the Engineering Societies of Western Europe and the U.S.A., Paris.

September 9-15. 124th National Meeting of the American Meteorological Society, Toronto, Canada.

September 10-14. Main meeting of the Association of German Smelters and Mining Personnel, Bonn.

September 12-18. Third Biennial Congress of the International Union of Leather Chemists Societies, Barcelona.

September 13-25. First International Instrument Congress and Exposition of the Instrument Society of America, Philadelphia.

September 14-16. Meeting of the Technical Board of the International Institute of Refrigeration, Baden-Baden and Karlsruhe.

September 14-16. Meeting of Commission No. 9: Education, International Institute of Refrigeration, Baden-Baden and Karlsruhe.

September 14-19. General meeting in conjunction with commemoration of 100th Anniversary of birth of Wilhelm Ostwald, Society of German Chemists and German Chemical Society, Hamburg.

September 21-25. Autumn Meeting of the Institute of Metals, Southport, Lancs.

CHEMICAL & PROCESS ENGINEERING ENQUIRY BUREAU

Stratford House, 9 Eden Street, London, N.W.1

Readers requiring names of suppliers of raw materials, machinery or finished products should complete this form and attach it to their business note-heading. Please state approximate quantities required.

We wish to know names and addresses of suppliers of the following:

For office use only. No.

Date

